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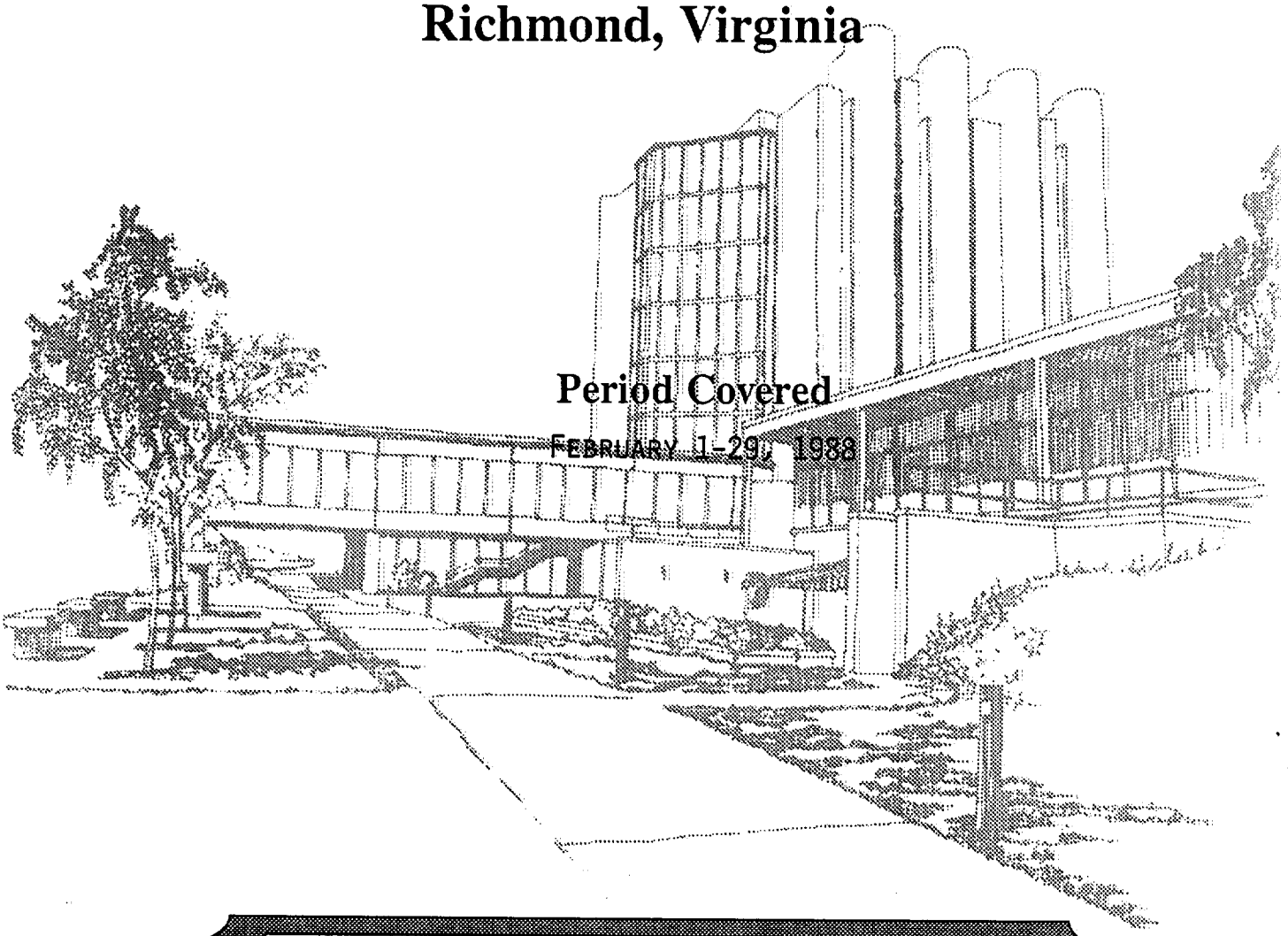

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PROJECT NUMBER: 2100
PROJECT TITLE: New Product Innovations
PROJECT LEADER: C. J. Campbell
PERIOD COVERED: February, 1988

I. PROJECT HUMIDOR

A. **Objective**: Develop a moisture release device for use inside a cigarette pack which will maintain the Pack OV at the desired level.

B. **Status/Plans**: A Focus Group test was conducted February 2-4 in Arizona, New Mexico, and Colorado. The concept and samples were generally well received by most of the subjects and were perceived as providing a benefit to the customer.

A project update was presented to the N.Y. Marketing Group on Feb. 25 and direction was received to pursue this project with Engineering. Production equipment will be set up to produce this pack for further testing. We will also begin investigation on use with menthol and soft pack.

II. PROJECT YOUTH

A. **Objective**: Develop a means for maintaining fresh cigarette flavor in a hermetically sealed pack.

B. **Status/Plans**: Ageing of irradiated cigarette is complete. Cigarettes have been tested analytically and subjectively to determine the effectiveness of irradiation in controlling off flavor formation in a hermetically sealed pack. The results of these tests will now be summarized to determine future direction.

III. FLAVOR ENCAPSULATION

A. **Objective**: Develop the means to encapsulate flavors for use in new or improved products.

B. **Status/Plans**: Initial testing of a gelatin walled capsule containing peppermint flavor from Dodge was successfully completed. Additional environmental testing and use testing will determine if the gelatin material is acceptable instead of urea-formaldehyde. An acceptable adhesive will also need to be found.

IV. EMBOSSING CONCEPTS

A. **Objective**: Explore the potential advantages of embossing cigarette paper, tipping and plug wrap for both cost savings and aesthetic appeal.

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B. **Status/Plans**: Efforts to produce test cigarettes on a machine from embossed materials are continuing. Three samples of embossed cigarette wrapper were completed and will now be evaluated for weight savings and CO reduction. Bobbins of plugwrap embossed to several porosity levels were produced, but attempts to make them into cigarettes for evaluation have not been successful.

The initial test of an embossed spacer for Virginia Slims cartons is complete and results are promising. Further testing, including ship testing, will now follow.

V. TAMPER EVIDENT PACK

A. **Objective**: Develop potential packaging materials and processes for positively and obviously indicating an attempt to tamper with a pack.

B. **Status/Plans**: A proposal from a second consultant to develop ideas for constructing tamper evident packaging is now being considered.

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PROJECT NUMBER: 2105
PROJECT TITLE: Filter Development
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: February, 1988

I. FILTER MATERIALS, LTD.

A. **Objective**: Develop polypropylene tow that is equivalent to cellulose acetate.

B. **Results**: Two mapping studies were completed on FML models at 3mg and 6mg. The data shows a significant difference in liking of the 6mg model compared to other Ultra Low Delivery cigarettes with the exception of Merit Ultra Lights

Further analysis of smoke chemistry on various FML filter models has shown that the reduction of wax and addition of triacetin and lubricant slightly reduces the differences in filtration of FML filters compared to CA filters.

C. **Plans**: Various filter additives will be tested for effects on smoke chemistry. Data from subjective testing will be reviewed to establish further test plans.

II. NEW FILTER CONCEPTS

A. **Objective**: Develop unique products using novel filters.

B. **Results**:

Modified Plastic Filter: E.E.M.A. region personnel requested a modified filter design having a minimum dilution to the smoking machine of 30%. This will be achieved by adding a third row of perforation between the flute blockage and the cigarette rod.

PVA Matrix: A PVA Matrix has been applied to non-porous plugwrap which complicates addition of dilution. Evaluations were conducted with a range of porous plugwraps. Bleed through of the PVA was not observed with plugwraps having a porosity below 7000 Coresta. Typically, the application did reduce porosity by one half.

C. **Plans**: Previous Modified Plastic Filter processing has been done with continuous adhesive application. Patterned application is now being evaluated. The PVA Matrix aging study will be completed in May.

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PROJECT NUMBER: 2304
PROJECT TITLE: Flavor Development - New Products
PROJECT LEADER: Barbara G. Taylor
PERIOD COVERED: February, 1988

I. STUDIO

A. **Objective**: To develop subjectively acceptable cigarettes with reduced sidestream visibility with flavor characteristics similar to Marlboro Lights.

B. **Status**: Trim II - The objective is to develop an ultraslim product with 30% reduction in sidestream visibility when compared to Capri using calcium carbonate paper. Models were evaluated with cigarette papers containing 35% calcium carbonate and various additives (succinate, MAP, and CMC) to effect burn rate and ash appearance. From preliminary results, the model containing 6% succinate had the best ash appearance and least objectionable off-taste, however, the product had a 6.9 puff count.

II. FILTER ADDITIVES

A. **Objective**: A. Evaluate the effects of filter additives on the subjective characteristics of cigarette smoke (evaluations on blends and blend components. B. Use filter additives to modify and enhance the most desirable smoke characteristics.

B. **Status**: GCC blend cigarette models were made using the filters containing various levels of citric acid (1.8, 1.2 and 0.6mg) in 19mm CA plug. Once the analytical data is available on these models, subjective evaluations will be done. The 11mg tar models that used tipping papers coated with citric acid, potassium phosphate, and potassium citrate were subjectively evaluated. The test models were different from the control and each other. Samples of these models were also mentholated by foil for subjective evaluations.

III. SWEET FLAVORS

A. **Objective**: To create/develop flavors that provide a sweet and/or coumarin-type character in smoke.

B. **Status**: CR 2643 was evaluated subjectively on an 11mg tar cigarette (no aftercut). At 1ppm, it presented a sweet, powdery, coconut-like character to the smoke. Further evaluations are being done with this product.

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PROJECT NUMBER: 2305
PROJECT TITLE: Flavor Development-Brand Modification-Menthol/Distinctive
PROJECT LEADER: H. M. Maxwell
PERIOD COVERED: February, 1988

I. ALTERNATIVE FLAVORS:

A. **Objective**: To identify and qualify alternative sources for several flavoring materials.

B. **Status**: Two alternate vendors of block licorice are currently being considered. Semi-works cigarettes were produced and rejected due to CI analytical results. They will be remade.

C. **Plans**:

Cigarette Remake	March, 1988

II. PROJECT MOOG:

A. **Objective**: To develop the expertise to produce cigarettes that are subjectively equivalent to Salem, Newport and Kool.

B. **Status**: The N-type POL's were remade and are in testing. Due to POL anomalies, K-type cigarettes are being reviewed. Semi-works cigarettes were requested to review blend subjectives and flavor modification, if necessary.

C. **Plans**:

N-type POL Results	March, 1988
K-type Semi-works Cigarettes	March, 1988

III. PROJECT FLEX:

A. **Objective**: For reasons of flexibility, economics and logistics, alternatives for fifteen PM flavors are being identified.

B. **Status**: Merit retesting, (POL and Walk in panel), is in progress. POL testing on Marlboro has been requested. Qualification of the remaining Flex flavors is in progress. Identification of replacements for some internationally procured Flex flavors is continuing. These samples are being accumulated by Operations Services.

C. **Plans**:

Complete Flex Flavors	March, 1988

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Complete Internationally Procured Flex Flavors	Indefinite
Replicate Merit POL	April, 1988
Marlboro POL	To Be Scheduled

IV. **PROJECT TRIM**:

A. **Objective**: To develop ultra slim product candidates with 17mm circumference.

B. **Status**: Assistance was rendered to Project Trim. Semi-works "Bond" type cigarettes were evaluated and found to be worthy of further testing. Menthol and nonmenthol cigarettes were produced for Ad/Pack testing.

V. **PROJECT LEVO**:

A. **Objective**: To determine the subjective differences between 100% Natural and 100% Synthetic menthol and to develop flavors to make them subjectively equivalent so that manufacturing would have complete flexibility to use them at any ratio.

B. **Status**: POL retesting of 100% natural vs. 100% synthetic menthol is scheduled. In-house testing is also scheduled.

Cigarettes for vendor qualification are being tested on the walk-in panel.

C. **Plans**:

POL Remake	March, 1988
Evaluation of Vendor Qualification Cigarettes	February/March, 1988

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PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization and International Support
PROJECT LEADER: J. L. Spruill
PERIOD COVERED: February, 1988

I. MARLBORO STANDARDIZATION

A. **Objective**: Analytical and subjective evaluations of production Marlboro KS/LS.

B. **Status**: January 7 production pick-up completed analytically and subjectively. A February 17 factory pick-up has been received.

C. **Plans**: Subjective and analytical evaluations of February 17 pick-up.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

A. **Objective**: To provide subjective direction for programs within R&D and manufacturing locations.

B. **Status**: Twenty-three panels completed during the reporting period.

C. **Plans**: Provide assistance as needed.

III. PROJECT NATURAL

A. **Objective**: To develop an 85 mm full-flavored prototype using blend components and flavor systems which will provide a natural blended product.

B. **Status**: 85 mm and 100 mm No-Additive blend models achieved analytical targets. Recessed filter models using no-additive blends and flavored blends will be made first week in March.

C. **Plans**: Analytical and subjective evaluations of recessed filter models. Fabrication of No-Additive blend models for ad-pack tests.

IV. FLAVOR RESOURCE DATA BANK

A. **Objective**: Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.

B. **Status**: Fifteen flavor vendor samples received for evaluation.

C. **Plans**: Sample evaluations and division updated with information.

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V. INTERNATIONAL SUPPORT

International Brands Smoking Panel

A. **Objective**: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.

B. **Status**: Samples profiled from Thailand, Japan and Korea.

C. **Plans**: Provide assistance as needed.

PROJECT ALPINE

A. **Objective**: Develop a recessed filtered menthol product for Singapore.

B. **Status**: Model ready for PMI testing.

C. **Plans**: Await results.

PROJECT STARSHIP

A. **Objective**: Develop a 12 mg Chesterfield for Japan.

B. **Status**: L&M Milds now in production at Cabarrus.

C. **Plans**: Subjective evaluations of production samples.

PROJECT MERIT/GALAXY

A. **Objective**: To create and develop an 85 mm Merit for Japan.

B. **Status**: Production cigarettes in cold storage.

C. **Plans**: No plans for launch.

PROJECT "DANNY"

A. **Objective**: To develop a cigarette for production in Malaysia with inclusion of up to 50% local tobacco subjectively comparable to U.S. Marlboro.

B. **Status**: Same.

C. **Plans**: Awaiting samples from Malaysia.

PROJECT LIGHT/ULTRA

A. **Objective**: Develop cigarette models at 4, 6 and 8 mg with a new blend and flavor system.

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B. **Status**: Four models requested and made using "Target" blend, two casing systems and four aftercuts.

C. **Plans**: Evaluate models.

PROJECT SWEET

A. **Objective**: Development of a distinctively sweet cigarette for the Japanese Market.

B. **Status**: Two models have been requested using two blends but with the same total blend casing and aftercut system. Each models will be made at 8 mg with 21 mm charcoal filter and at 6 mg with 27 mm charcoal filter.

C. **Plans**: Evaluate models for possible testing.

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PROJECT NUMBER: 2307
PROJECT TITLE: Flavor Investigation/Nonvolatile Flavor Investigation/Processed Tobacco
PROJECT LEADER: W. R. Raymond
PERIOD COVERED: February, 1988

I. FLAVOR INVESTIGATION:

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. A method has been developed using multidimensional GC to quantitatively determine vanillin and ethyl vanillin on filler and in finished cigarettes. Refinements are in progress. The ability to determine vanillin and ethyl vanillin will provide corroborating data for the anethole method currently in use to determine A/C application levels. Determination of vanillin and ethyl vanillin in addition to anethole also should be of use in monitoring correctness of A/C preparation. The method has been tested with MF 85's and PM Blues.

2. Headspace analyses have been performed to compare A/C levels in MF Lts. Menthol cigarettes manufactured at SS and MC. Additional testing was performed of SS cigarettes made from SS- versus MC-produced filler. Filler samples taken throughout the MC and SS cigarette manufacturing processes also were examined to identify points of flavor loss. Results are undergoing evaluation.

3. Analyses of flavors and flavor ingredients included:

   a. Compositional comparison of four natural rum flavors;
   b. GC profiling and MS identification of components of three natural/artificial rum flavors and two samples of 04-257;
   c. Purity determinations for 20 flavor chemicals;
   d. Alcohol, water, PG, glycerine, anethole and vanillin analyses of MF A/C's and preblends for Project Grain;
   e. GC profiling of current A/C formulations for MF, Merit, B&H, New MF Menthol and Parliament;
   f. Vanillin, PG and glycerin analyses of a casing flavor for ART;
   g. GC analyses of three flavors undergoing storage stability testing.

4. Nontobacco materials analyses included:

   a. Headspace-GC analysis of MUL (new graphics) cartons, boxes and cigarettes to determine volatile transfer from packaging to product;
   b. Headspace-GC analysis of Merit tipping paper;
   c. Headspace-GC analysis of L&M Milds labels.
   d. Headspace-GC analysis of plastic boxes for Project FOX.

2022162284

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II. NONVOLATILE FLAVOR INVESTIGATION:

A. **Objective**: To investigate tobacco-natural, tobacco-identical and other nonvolatile fractions and constituents for potential application as cigarette flavor additives.

B. **Results**:

No activity this month.

III. PROCESSED TOBACCO:

A. **Objective**: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.

B. **Results**:

1. Analytical and subjective evaluations continued of roasted JONEX from three suppliers in order to develop purchasing and Q.A. specifications. Since results with the roasted extracts have shown considerable compositional and subjective variability, evaluation of native extracts is in progress to determine whether variability is a function of raw material or of processing.

2. Pilot RLB trials have been completed using dry flavor replacement materials from the various suppliers. Subjective selection of alternate flavors will be completed in March and followed by extended Park 500 replacement tests to confirm processing advantages.

3. Physical, chemical and subjective testing have been completed with ultrasonically-processed RCB. Analytical and subjective testing of control versus test RCB at standard blend levels in Marlboro cigarettes produced at SS showed no differences.

4. Evaluation continued with post-ART stem (both by direct incorporation in filler and substitution for stem in RL's). ART pilot trials with CRS versus FSS are being evaluated with DL filler. Subjective and analytical differences are being compared between top and bottom stem baskets. CRS from multiple ART runs is to be blended for IS trials at LVL in mid-March to test utilization in Marlboro and PMSL.

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PROJECT NUMBER: 4009
PROJECT TITLE: Development Smoke Studies
PROJECT LEADER: B. L. Goodman
PERIOD COVERED: February, 1988

I. PROJECT STUDIO

A. **Objective**: Develop subjectively acceptable cigarettes with reduced sidestream visibility.

B. **Status**: Concentrated effort has been dedicated to develop an acceptable cigarette in the Trim configuration, using small particle size $CaCO_3$ wrapper as a sidestream reducing mechanism. More than twenty different paper variations (of Succinate, CMC, and MAP additive levels) have been evaluated for subjective acceptance, ash appearance, visible sidestream reduction, and analytical delivery. Trim II and Trim III blends have been utilized in developing a subjectively acceptable cigarette with the 100% small particle size CaCO3 paper from Kimberly-Clark. The most viable option to date incorporates 2-6% Potassium Succinate, approximately 1% CMC and 0% MAP, applied by KC at an adjusted pH. Further blend and flavor development will continue to optimize subjective acceptability.

The effect of a dual carbon/acetate filter is being evaluated on several magnesium hydroxide programs in an attempt to improve the subjective response of reduced sidestream products. The amount of carbon loading in the filter and the subsequent effect on taste is also being investigated.

The Flavor Low 100mm low sidestream prototype with a CA filter is currently being evaluated on the Factory Panel versus Marlboro Lts. 100's

POL Mapping studies of two Flavor Low models have been closed out with results pending.

A partially expanded blend (expanded to 325 degrees) is being evaluated in the 70% reduction program with 35 % magnesium hydroxide wrapper.

Cigarettes of various levels of visible sidestream reduction will be evaluated visually on the O/C Panel, at both regular and Ultra Slim configurations in order to begin assessing the impact of low sidestream products on smokers and nonsmokers.

Plans: Evaluate blend and flavor modifications on TRIM with K-C's 100% Calcium Carbonate wrapper to improve subjective acceptance.

Continue development of perforated 35% $Mg(OH)_2$ prototypes with regular circumference using expanded blends.

2022162286

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PROJECT NUMBER: 2304, 2305, 4015
PROJECT TITLE: New Product Development
PROJECT LEADER: B. G. Taylor, H. Maxwell, and W. G. Houck
PERIOD COVERED: February, 1988

I. MARLBORO/MARLBORO LIGHTS MENTHOL

A. **Objective**: (1) Develop Marlboro Menthol to appeal more to Salem smokers. (2) Develop a Marlboro Lights Menthol brand extension to appeal to Salem Lights smokers.

B. **Status**: Retail sales in progress since 2/1/88. Replicate POL and an HTI testing being remade. Plans call for shipping 4/1/88. Recent study results show promising areas for improving and stabilizing menthol-in-smoke deliveries. These include menthol blockers (carbowax), heat setting of highly mentholated filters before tobacco rod attachment, and supplemental menthol release during heat stress via the PVA encapsulation mechanism.

C. **Plans**:

Complete replicate POL's 2148, 2149 and HTI 1719	April/May '88
Monitor National Prod. Start-up (includes field pickups)	On-going
Special Studies	On-going

II. MARLBORO ULTRA LIGHTS

A. **Objective**: To develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.

B. **Status**: POL testing scheduled versus MUL 85 and 100. First make was unacceptable subjectively. Remake in progress.

C. **Plans**:

Complete POL testing with MUL	April '88
Test Market	4th Qtr.'88

III. PROJECT TRIM (Ultra Slim)

A. **Objective**: To develop 17 mm circumference/100 mm Ultra Slim product candidates.

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B. **Status**: POL testing with "milder" Trim candidates completed with satisfactory results. POL testing in progress with menthol and nonmenthol versions of a modified blend, casing, and A/C system. Ad/pack testing in progress with both menthol and nonmentholversions of the modified blend, casing, and A/C system. Test marketing of an ultra slim candidate has been delayed in order to incorporate low sidestream cigarette paper into the development Trim II). Candidates using the current filler and flavor systems are being developed. POL testing is planned.

C. **Plans**:

Current POL testing Reg. and Men.	Completed
Factory trial (Louisville)	
Trim I	Completed
Trim II - low sidestream	4/88
Factory start-up - Trim II	5/88
Test Market (Retail) - Trim II	7/88

IV. PROJECT ART

A. **Objective**: To investigate methods to decrease the nicotine content in tobacco. To develop full-flavored, ultra low, and light products using the low nicotine tobacco products.

B. **Status**: Prototype development continues with emphasis on 8, 11, and 16 mg. Regular and Menthol models - 85 and 100 mm. Product development emphasis is on Merit and Marlboro Lights prototypes. Extended use testing in progress with nicotine add-back. Blend/blend component and AB level studies in progress. Product development direction/recommendations being formulated.

C. **Plans**:

Blend development/evaluation	On-going
Product/flavor development	On-going
Product development recommendations	1st Qtr. '88
Test Market	4th Qtr. '88

V. PROJECT EXTRA

A. **Objective**: The development of 4-8 mg product candidates that have the subject taste of product with twice the tar.

B. **Status**: Mapping studies complete with paper/CA candidate. CR chemical research and evaluation continues for possible new/enhanced flavor systems. Filter flavor work continues to increase flavor impact and/or modify existing flavors. Low density cigarette models made using ART filler completed. Puff-by-puff profiles in progress with Extra prototypes and commercial brands.

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C. **Plans**:

Sensory Mapping	On-going
Flavor development/screening	On-going
Prototype Development/evaluation	On-going

VI. ALPINE

A. **Objective**: To develop generic-value entry brand extensions of the Alpine menthol cigarettes in KS and 100 mm full flavor and lights versions.

B. **Status**: The specifications have been generated using Marlboro Menthol full flavor and lights with mentholated foil as a guideline. MB-4 blend will be used for initial factory start-up. The specification for the 100 mm full flavor cigarette is a new cigarette design.

C. **Plans**:

Factory start-up	week of 3/7/88
National introduction	April 1, 1988
Blend development (replace MB-4 with low-cost blend)	On-going

2022162289

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PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development
PROJECT LEADER: L. Mims Evans, R. E. Tinker
PERIOD COVERED: February, 1988
WRITTEN BY: A. H. Confer

I. PROJECT DORA (HONG KONG)

A. **Objective**: Develop an 11.5 mg KS product in both charcoal and non-charcoal versions.

B. **Results**: PMI testing indicated a directional preference for the charcoal version.

C. **Plans**: An April launch is planned, and product specifications are being finalized.

II. PROJECT RFM (SINGAPORE)

A. **Objective**: Develop a subjectively acceptable menthol product with a recessed filter.

B. **Results**: A 10 mg prototype and Salem ex Singapore have been ringtipped, analytically and subjectively approved, and shipped for PMI testing.

C. **Plans**: Consumer testing will take place March/April.

III. PROJECT NOVA (VENEZUELA)

A. **Objective**: Develop a 70 mm, 22 mm circumference cigarette.

B. **Results**: Development of the full delivery product has been completed.

C. **Plans**: A "lights" version has been requested with a nicotine delivery of 0.6 - 0.7 mg/cigt. Specifications are to be finalized in May, with launch targeted for 3rd quarter.

IV. PROJECT FAMOUS (VARIOUS COUNTRIES)

A. **Objective**: Optimize Chesterfield as a worldwide competitor to Camel.

B. **Results**: The Development Team met and meeting minutes were published. A series of prototypes was prepared in Neuchatel.

C. **Plans**: The prototypes will be evaluated in mid-March, and consumer testing of the selected prototype(s) vs. Swiss Camel will begin in mid-April.

2022162290

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PROJECT NUMBER: 4024
PROJECT TITLE: Japan Product Development
PROJECT LEADER: C. B. Altizer
PERIOD COVERED: February, 1988

I. LARK OPTIMIZATION

A. **Objective**: To optimize the subjective acceptance of the Lark family while retaining the American blended cigarette character.

B. **Results**: Prototypes of the Lark 80 mm box product, both Full Flavor and Milds, were evaluated and determined to be subjectively acceptable by the Japan Panel. The necessary information for start-up of this line extension was given to Operations Services.

Cigarettes are being prepared for PMI and Danchi tests of Lark and Lark Milds with an expanded tobacco substitution, as well as, certain grade substitutions necessary for brand maintenance.

C. **Plans**: Further development work will be based on Danchi and PMI test results.

II. PROJECT STARSHIP

A. **Objective**: Develop a product in conjunction with a Young American Image, that would compete successfully with latest generation of U.S. imports.

B. **Results**: Results of the Danchi Panel Tests of Starship models against both Kent Milds and Lucky Strike showed little differences between the cigarettes, but all the Starship models tested well. Generally, though there were no statistically significant differences in liking scores, the Kent type product was perceived as being strongest and least liked while the L&M type was liked best among the Starship models. Based on these results a recommendation was made and accepted to use the Starship model with L&M casings and flavors as the L&M Milds products due to the high liking scores and its attractiveness to Mild Seven smokers.

A production trial for L&M Milds was done in Cabarrus. The trial model was analytically and subjectively approved for production startup. Production began in late February.

C. **Plans**: Launch project Starship as L&M Milds, April 1 in Japan.

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Source: https://www.industrydocuments.ucsf.edu/docs/xnxm0000

PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: February, 1988

PACKAGING STUDIES

A. **Objective**: Assist New Products Directorate in evaluating new packaging concepts and products, and provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance.

B. **Status/Plans**:

1. **Project Fox**: Currently awaiting pigmented versions of the test sample found to be acceptable by subjective smoking.

2. **Project Fresh**: A six week Accelerated Aging study of the current Ad/Pack production was completed. A report is in progress.

3. **Cigarette Cold Storage**: At their request, we provided the Transportation Department with an explanation of the tempering process, necessary when removing cigarettes from cold conditions for shipment, and a list of facility attributes that must be considered when looking for additional cold storage locations.

4. **Foil Carton Overwrap (Reynolds)**: Water Vapor Transmission Rate (WVTR) tests found the current foil overwrap (tissue backing) to provide a water vapor barrier 461% greater than this modified material (bond paper backing). Since this change should not affect WVTR, Reynolds will be asked to scrutinize incorporated process changes. A 10% cost savings on foil carton overwrap could be realized if this material were to provide comparable product protection.

5. **Hoechst/Celanese Polypropylene (Mexico)**: Cigarettes packed using the Hoechst/Celanese polypropylene failed subjective smoking evaluations. After QA (Microstructure) analysis found no difference in material make-up that would account for subjective failure, we were told that this particular bobbin was shipped to us in a 'green' wooden box and may have been contaminated. Samples packaged with a new bobbin of material have been aged and submitted for subjective smoking evaluation.

6. **Taste, Odor, Stale Customer Complaints**: Marlboro KS SP conditioned to OV levels of 10%, 12%, 13%, and 14% were supplied to PED for testing on the M/C panel.

7. **Strip Laminated Foil**: A six week storage study was completed to evaluate this material as a possible means of reducing foil cost on generics. The material is undesirable as staining penetrates the SP label, in the area where no foil is present on the liner, and becomes visible on the outside of the package.

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Source: https://www.industrydocuments.ucsf.edu/docs/xnxm0000

PROJECT NUMBER: 0400
PROJECT TITLE: Low Density Rod Development
PROJECT LEADER: R. S. Mullins
PERIOD COVERED: February, 1988

LOW DENSITY ROD

A. **Objective**: Develop a continuous process for the production of reduced density cigarettes.

B. **Results**: Additional testing of the modified microwave cavity with the alumina pass tube was conducted. The application of vacuum at either end of the tube was found to be unsatisfactory as it resulted in excessive vibration of the tube belt within the cavity. Applying compressed air to the cavity and allowing the air to exit through the pass tube, via the slot in the tube, prevented the build-up of filler particles but did not prevent the build-up of condensed tobacco volatiles on the wall of the tube. The standard cavity, without a pass tube, was subsequently modified to increase the air flow through it by perforating a section of waveguide to allow air to more easily enter the cavity and by increasing the diameter of the holes through which suction is applied to the cavity. The increased air flow produced by these modifications greatly reduced filler build-up without any apparent condensation of tobacco volatiles. Possible additional cavity improvements and alternate designs will be discussed on March 9 with Charles Buffler, a microwave consultant associated with General Foods.

Results from the Dextran evaluation indicated that increasing the binder level from 6% to 8% improved firmness but further increasing the level to 10% provided no additional improvement. Even at the 8% level, however, the Dextran continued to produce less firm cigarettes than did pectin. Significantly more filler degradation was observed in this test in the coating operation using uncased all-lamina filler than was observed in the previous test using cased Marlboro filler without ET, ES, or aftercut. Since application of the Dextran requires only about 5 minutes, this indicates that most of the degradation of the uncased all-lamina filler is occurring very rapidly at the beginning of the binder application procedure and is further evidence that a casing system is needed for the all-lamina blend being used in the low density program.

No significant differences were detected between the physical quality of cigarettes produced with pectin having a 40% degree of esterification (DE) and that of cigarettes produced with the normal pectin having a 65% DE. As reported last month, however, the binding obtained with pectin having only a 4% DE was so poor that no cigarettes could be produced with it in the low density process. The coating obtained with the 40% DE pectin resembled that obtained with the 60% DE pectin at addition levels of 6% but did not appear as consistent or smooth at addition levels of 9% and 12%. The coating obtained with the 4% DE appeared very variable and had many more tendrils extending from the shred surfaces.

Production of cigarettes for the study to determine the effect of molecular weight on the performance of Genu pectin has been completed and the cigarettes submitted for testing. Evaluations of low density

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samples produced for sidestream studies have not yet been completed. Maker modifications completed this month included installation of a motor and drive unit for the carding drums, a fan for the overhead transfer belt, and an improved steaming garniture.

C. **Plans**: During the week of March 14, produce cigarettes for an investigation of the effects of density, blend, cut width, paper weight, paper porosity, and paper additive on the physical, smoking, and subjective characteristics of low density cigarettes. Compile and evaluate data from the molecular weight study. Conduct preliminary evaluations of binder application to filler at the maker. Begin production of cigarettes for an evaluation of the effects of filler CV, OV, and size on the firmness of bound rods.

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PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: February, 1988

I. IMPROVED SHEET PROPERTIES

A. **Objective**: Improve the physical characteristics and blend performance of reconstituted sheet materials.

B. **Results**:

1. ART Project - ART #54 and #57 shredded stems (thin lamina filler extraction) incorporated into RL, as well as direct inclusion models of #57 and #59 (CRS/cut strip filler), gave a more intense hot/harsh subjective response in Marlboro blends than have previous ART stems. Current efforts are focused on the evaluation of ART #87 and #88 CRS (from the extraction of the DL lamina blend) via both direct inclusion and incorporation in RL.

   A program has been initiated to evaluate the microbial storage stability of pre-ART and post-ART stems.

   A second Semiworks study is in progress to determine cigarette-to-cigarette blend variation with ES, IS and shredded stems using ethyl paraben as a tracer. This will be completed in March.

   Pilot and handsheet studies with added citrate indicate that incorporating ART stems in RL will give a 10% loss in tensile strength. Drainage characteristics emulate a more refined stock, i.e., drainage rates are lower but couch roll vacuum, and therefore sheet dryness, are higher. These effects are equivalent to a 1-2% increase in baseweb solubles.

2. Humectants - Glycerin-free RCB test sheets, including versions with 2% and 4% PG plus controls, were remade at the BL Plant. Machine-made 100% cigarettes are being subjectively screened.

   Production RCB and RL test sheets containing potassium propyl paraben were judged subjectively equivalent to controls. The production of PG/glycerin-free sheet materials using isosweet and potassium propyl paraben are tentatively scheduled for the end of March.

3. RL - Quaker #2052 Yankee dryer release agent (carbowax) has exhibited the best performance from both a process and subjective standpoint. Additional long term trials for subjective testing have been completed.

   Licorice extract added to pilot baseweb (0.5%) showed no improvement in size absorption. An increase in absorption had been indicated by handsheets.

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C. **Plans**:

1. Complete subjective comparison of ART CRS and SS (DL filler extraction) by direct stem inclusion and incorporation in RL.

2. Complete the cigarette-to-cigarette blend variation study with stem products.

II. **SUBJECTIVE MODIFICATION OF RL**

A. **Objective**: Improve or modify the subjective character of RL.

B. **Results**:

1. Pilot 150B sheets were completed to qualify alternate suppliers for dry flavor replacements. Subjective screening of 100% sheet cigarettes is completed; blended cigarettes are scheduled for Semiworks.

2. No additional POL testing will be conducted with the existing Park 500 Modified 150B test sheet. Efforts are directed at evaluation of ultrasonic devices to reduce the particle size of precipitated phosphates and eliminate size press nip rejection. If successful this would necessitate a demonstration trial at Park 500, with subsequent subjective evaluation of this new test sheet.

C. **Plans**:

1. Complete subjective blend evaluation to qualify alternate suppliers for dry flavor replacements.

2. Select ultrasonic equipment for pilot trials to reduce the particle size of phosphate precipitates.

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Source: https://www.industrydocuments.ucsf.edu/docs/xnxm0000

PROJECT NUMBER: 1313
PROJECT TITLE: Semiworks Process Development
PROJECT LEADER: W. G. Sanderson
PERIOD COVERED: February, 1988

A. **Objective**: Provide engineering and technical support to improve the performance and efficiency of the Semiworks operation.

B. **Results**:

Bright Flavor Flow System - Semiworks Primary (Oliver) - A manual detailing the operating procedures for the Bright Flavor Flow System in the Semiworks primary was issued. This manual contains operating standards, flow meter calibration procedures, cleaning requirements, system lay-out diagrams, and control logic schematics. Detailed operating procedures for training purposes are also included.

Evaluation of a 30 pound Capacity Cylinder (Nguyen) - The evaluation of a 30 lb. capacity cylinder for use in the Semiworks small scale primary operation was conducted. Results, from this evaluation, indicated that there was no significant difference between the control 15 lb. batch cylinder and the experimental 30 lb. capacity cylinder with regard to spray uniformity (PG & GLY) or blend uniformity (%RCB & %RL). Additional development on the 30 lb capacity cylinder will be conducted to determine the feasibility of using a stationary spray rack in place of the reciprocating spray arm.

On Line Laser Perforator For Product Development (Osmalov) - A Hauni on-line laser perforator was installed on the Semiworks complex 3 Mk-9/Max-S module for new product development work. The unit is configured to allow production of conventional parliament brands as well as new products with 5, 10, 15, and 20mm recessed filters.

Recessed Filter Development Work (Osmalov) - The reconfiguration of a filter combiner to produce a 15/16.5 plug with a 16.5mm recessed section was completed in the Semiworks make/pack area. Trial runs to check-out these modifications will being in early March.

C. **Plans**: Transfer the technology developed for the TLA primary process to the Semiworks Small Scale operation. Review all casing systems in the primary to provide improved flexibility and cleaning. Continue recessed filter development work and support for the on-line laser perforator for product development.

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PROJECT NUMBER: 1333
PROJECT TITLE: Semiworks Process Control
PROJECT LEADER: D. A. Phan
PERIOD COVERED: February, 1988

A. **Objective**: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. **Results**:

Bright Top Casing Flavor System Upgrade (Oliver) - Provox configuration for the second bright casing control system was complete. Instrumentation has also been received. Installation is planned to be completed by March 30.

Adt Dryer Control Improvements (Sims) - A special data logging and retrieval system will be designed to obtain accurate process data for all production runs through the dryer and to aid process analysis. System design is planned to begin on March 21 and be complete by April 15.

Tobacco Lot Analysis (Phan/Sims) - Assistance has been provided to PM Engineering to startup the TLA dryer and conditioner system at the QA Audit facility.

Semiworks Primary Computer Upgrade (Oliver/Sims) - Significant progress has been made to bring up both the host and backup computers on the Fisher Provox data highway. Application software is being modified and tested in the new system environment.

Semiworks Cigarette Defect Database (Sims) - A database was created in an IBM-PC for critical cigarette defects. Software has also been written to computerize the Semiworks make/pack QA monthly report.

Quality Assurance (Rowsey) - A purchase order was issued for an IMPS pack seal instrument to establish baseline performance for all Semiworks packers.

A Hauni Proscan rod analyzer was upgraded with improved software. Its operation has been reviewed with Hauni engineers. This instrument will be studied by Engineering Development before it is returned for use in Semiworks make/pack area.

C. **Plans**: Continue the work on the primary backup computer upgrade project. Complete the installation and checkout the second bright top casing flavor control system. Begin the design of a special data logging and retrieval system to support the Adt dryer improvement project. Continue to provide electrical plant engineering support to the Semiworks and conduct routine QA functions.

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PROJECT NUMBER: 1503
PROJECT TITLE: Modified Smoking Materials
PROJECT LEADER: J. G. Nepomuceno
PERIOD COVERED: February, 1988

I. FOAMED FILLER BINDER

A. **Objective**: To develop a process for applying a subjectively acceptable foamed binder to the tobacco filler during making to improve cigarette quality.

B. **Results**: Significant equilibrated firmness improvement (0.2 to 0.3 mm firmness units) was demonstrated on Cambridge cigarettes to which a foamed solution of maltodextrin binder (hydrolyzed starch) was applied. A formulation of 58% maltodextrin (with 3% licorice added as a foaming agent) was added to the cigarettes at addition rates of 0.2%, 0.5% and 0.8%. Follow-up tests are planned where the moisture content of the feed tobacco will be adjusted to compensate for the moisture from the binder. Using maltodextrin instead of degraded pectin is advantageous to the process as this binder can be applied at higher solids concentrations, thus minimizing the moisture increase due to the binder addition. Maltodextrins are also more readily available from the manufacturers than the degraded pectins and at a lower cost (≈$0.27/lb).

C. **Plans**: Do follow-up testing on the maltodextrin solutions using tobacco with the feed OV's adjusted for the moisture increase due to the binder addition. Complete evaluation of thermally degraded pectin solutions from Obipektin (Switzerland) using solution concentrations of 50% binder solids.

II. LOW DENSITY ROD (BINDER APPLICATION)

A. **Objective**: Produce pre-coated tobacco via the batch process to support the current product and process development efforts for the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be easily scaled up to commercial scales.

B. **Results**: Tobacco pre-coated with dry pectin powder (as opposed to pre-coating with a sprayed solution) was tested on the low density maker. Results showed that a significant portion of the powder was lost in the hopper, demonstrating a need to further improve the adhesion of binder to the tobacco. Pre-treatment of the tobacco with citric acid has been shown to improve this adhesion. Overspraying with water or a light coat of pectin solution will also be explored.

Installation of the process for continuously applying liquid binder solutions to the tobacco (coating reel) is nearing completion. Start-up of this process is expected within the coming week.

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C. **Plans**: Continue development of methods for pre-application of binders to the tobacco. Plans for evaluating use of the tower to dry the tobacco after binder spray application is under review. This work is another approach being considered as an alternative to the other methods currently under evaluation for applying binders to the tobacco.

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PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
SECTION LEADER: S. R. Wagoner
PERIOD COVERED: February, 1988

I. PROJECT ART - PILOT PLANT SUPPORT

A. **Objective**: To provide processes for converting and casing stem materials into useable form for the Bermuda Hundred Pilot Plant.

B. **Results**: Batches of shredded stems and rolled/cut stems were cased with monopotassium citrate and shipped to the Bermuda Hundred Plant. Stems were shredded on the D Pilot stem line for future ART runs. A conveyor was installed to feed the blending silo in D Primary, providing the capability of blending filler and stems within individual ART runs.

C. **Plans**: Continue to produce stem products as required by Project ART.

II. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

A. **Objective**: To conduct trials providing information for development of the ART commercial process.

B. **Results**: Trials were conducted to identify the optimum process for casing post-ART filler. In the first trial at the Semiworks, extracted filler was cased at the post-ART moisture of 25% and then dried to 12.5% in the Adt dryer. Even though the filler had been processed through the declumper at the Bermuda Hundred Pilot Plant, some pads survived through casing. For the next trial, the filler was dried to approximately 17% following extraction. This step appeared to eliminate the filler pads, such that there was no problem casing and drying the material. In addition, laboratory trials have shown that exposing post-ART filler to a steam atmosphere will loosen clumps before casing. Results to date indicate that the casing can be applied uniformly at ART extraction moistures, if the filler is properly loosened through a combination of mechanical and thermal treatments.

A trial evaluating two-step ammonium bicarbonate application was conducted for ART run 084. The DL strip blend at 14.3% OV (target = 16% OV) was initially cased with a 16.8% AB solution. Following cutting, the filler was cased at the Bermuda Hundred Pilot Plant with the same concentration of AB solution to yield a 26.7% OV and an AB level of 4.2%. The run achieved an extraction level of 98%. This method of AB application appears to be feasible, if the moisture is controlled properly.

Filler from ART run 080 was dried in the Sargent's tray dryer with air at 200F, and samples were taken with time for OV and AB analyses. The data indicated that the AB level was constant as the filler dried from 24 to 10% moisture. At 2.4% OV, there was a 22% reduction, and at an OV of 0.7%, the AB reduction was 44%.

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Thus, significantly reducing AB level by altering dryer conditions does not appear feasible.

To determine the subjective effects of ART processing, stems from ART run 086 were superheated in the 3" tower. This included uncased CRS feedstock, cased CRS that was dried at the Bermuda Hundred Pilot Plant, and cased CRS that was subjected to high pressure CO_2 and then dried. (Filler was not used in this run.) Evaluation indicated that the pre- and post-ART samples are subjectively more similar to one another than to the uncased CRS. Also, post-ART CRS and shredded stems from the top and bottom baskets of runs 087 and 088 were superheated for subjective evaluation. The segregated stems were blended for additional superheating runs and for sheet making trials.

C. **Plans**: Conduct tests utilizing the Quester steam tunnel to declump post-ART filler prior to casing.

Repeat testing of ammonium bicarbonate application on strip for commercial design.

Complete subjective evaluation of the above stem products. Conduct tests on the Louisville IS line to produce superheated IS from post-ART CRS.

Following installation, evaluate the Dickinson Admoist conditioning unit as an alternative to cylinders for conditioning whole stems prior to shredding or rolling/cutting operations.

III. BINDER DEVELOPMENT

A. **Objective**: Develop methods to produce binder systems for the foam bound rod, bonded ends, and low density rod programs.

B. **Results**: Assistance to Engineering's bonded ends evaluation was continued through the preparation of several sodium CMC solutions with the addition of licorice as a foaming agent. Also, solutions of sodium CMC with additions of Mg acetate and Ca acetate were prepared and characterized for evaluation on the R&D ink jet system.

Because of cost considerations, maltodextrin is being reevaluated as a foam binder candidate. Laboratory characterization (viscosity, foam quality, film strength, contact angle, surface tension) of maltodextrins at various dextrose equivalents was conducted to identify the optimum binder for process utilization. In addition, a degraded pectin solution was prepared for use in Engineering trials at FTR.

C. **Plans**: Continue to provide support as required to the above programs.

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IV. TMCI-ASTA SHEET

A. **Objective**: To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCI process and PM-RCB technology for international application.

B. **Results**: Microflake production was begun in the TSA Tarragona plant on the 22nd of February.

There have been indications during ASTA processing in Portugal and FTR that particle size was a factor influencing sheet quality. Some earlier work with CTB slurries also indicated that the type of grinding mill used for particle size reduction affected slurry viscosity possibly due to a particle shape factor. For these reasons, bright tobacco stems were ground in the laboratories of C. E. Raymond, Chicago, to three different sizes on both an impact mill and a roller mill. The mean particle size of these products was 120μ, 60μ and 25μ representing BL production dust, FTR ASTA feedstock and Portugese ASTA feedstock, respectively. This work may allow the total solids of the slurry to be increased and thereby reduce drying costs.

C. **Plans**:

1. Inspect Microflake production in Portugal and Spain during the week of 3/7/88 to make final arrangements for ASTA trials in Spain beginning 4/18/88.

2. Evaluate the effect of particle size and shape on slurry viscosity and the sheet quality of ASTA and RCB.

V. CTB

A. **Objective**: To design and build an equipment package for production of CTB in South American countries.

B. **Results**: The trials to evaluate equipment for production of CTB in Ecuador using Ecuadorian CT and burley casing were completed. The same concentration of CT in Marlboro burley casing was found to give the same slurry viscosity as US-CTB, but an excess of 50% CT in Lark casing was found to be possible for the same viscosity as US-CTB.

C. **Plans**: Ship equipment to Ecuador after some minor mechanical modifications are completed.

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Source: https://www.industrydocuments.ucsf.edu/docs/xnxm0000

PROJECT TITLE: Entomological Research
PROJECT NUMBER: 1101
PROJECT LEADER: D. L. Faustini
WRITTEN BY: R. M. Lehman
PERIOD COVERED: February, 1988

I. CIGARETTE BEETLE (CB) PHYSIOLOGICAL STUDIES

A. **Objective**: Conduct studies to determine methods for controlling CB infestations in stored tobacco using methoprene on the container surfaces.

B. **Results**: Cardboard cases, proportionately reduced to laboratory size, were treated on the inner surface with 500 and 5000 ppm methoprene. After CB infestation and storage in the laboratory for seven months the cases were opened and evaluated. The control was heavily damaged and an average of 2833 CB's per case were found to be present. In comparison the cases with 500 ppm methoprene averaged 172 CB's/case (light damage to product), and the cases with 5000 ppm methoprene showed very light product damage with only 11 CB's/case.

C. **Plans**: Write a special report.

II. METHOPRENE-TREATED FILM

A. **Objective**: Evaluate methoprene treated film for controlling CB's.

B. **Results**: A report was written summarizing the studies of methoprene-treated films as an ovicide.

III. AGELESS® OXYGEN ABSORBER

A. **Objective**: Determine dose response levels for effective use of two Ageless® (iron oxide) products against the CB.

B. **Results**: Laboratory studies have been initiated.

C. **Plans**: Complete studies and evaluate data by the end of March.

IV. STUDY OF CB INFESTATIONS

A. **Objective**: Determine the origins of CB infestations in cured tobacco.

B. **Results**: Consultations were held within PM and at Universal Leaf regarding monitoring programs.

C. **Plans**: Initiate monitoring program at ULT's Danville facilities.

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V. GAS FLUSHING

A. **Objective**: Evaluate gas flushing as a means to gather data for inert atmosphere chamber certification.

B. **Results**: Equipment has been installed and laboratory studies have been initiated.

C. **Plans**: Evaluate gas mixture equivalent to that produced for inert chambers which are to be used for the treatment of export cut filler.

VI. REFERENCES

1. Lehman, R. M. Notebook No. 8519, pp. 7-9, 13-14.

2. Minor, M. F. Matters on the methoprene-treated film report. Memo to D. L. Faustini; in press.

3. Lehman, R. M. Notebook NO. 8519, pp. 51-52, 54.

2022162305

Source: https://www.industrydocuments.ucsf.edu/docs/xnxm0000

PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: February, 1988

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

A. **Objective**: To develop methods by which to objectively and reliably evaluate human responses to cigarettes, smoke constituents and tobacco flavorants.

B. **Results**:

1. Cognitive NERP Study

The first phase of the modifications to the olfactometer are nearing completion. Contamination problems as well as a problem in the switching mechanism of the olfactometer have delayed the testing of the new odorant delivery system. These problems are currently being corrected and experiments to test the operation of the new system should begin the first week in March.

R. Greene has written a RPL procedure that allows the NERP waveform data to be automatically read into RS1. Investigation is underway to determine whether the current format of the data is suitable for conducting various multivariate statistical analyses available through BMDP. Additionally, PC statistical/graphical software packages are being evaluated for their utility in manipulating and displaying multivariate data.

C. **Plans**: Upon completion of the modification and testing of the olfactometer, plans are to continue parametric manipulations of the cognitive paradigm in order to determine the optimal parameters for elicitation of the late positivity of the NERP.

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PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: February, 1988

I. DIELECTRIC SPECTROSCOPY OF TOBACCO (D. Lowitz)

A. **Objective**: Develop improved process monitoring techniques.

B. **Results**: A report was issued detailing the correlation of the dielectric spectra of differently processed portions of a tobacco sample. Different portions of the same tobacco sample were found to have different CV values after being equilibrated to the same OV.

An invention record was submitted for the simultaneous monitoring of tobacco OV and dielectric spectrum. The technique can be used to reduce dust generation in the stemmery and to improve filler filling power in the Primary.

C. **References**: D. A. Lowitz, M. E. Counts, J. C. Crump, "Dielectric Analysis of the Tobacco/Water Matrix - Effect of Processing Route on Significant Tobacco Properties", Special Report #88-013, February 10, 1988.

II. OPTICAL PACK INSPECTION, EXPERIMENTAL (C. Harward)

A. **Objective:** Evaluate the Global Holonetics SMART CAMERA. Determine its effectiveness in discriminating between good and defective packs.

B. **Results**: The new, modified SMART CAMERA was received from Global Holonetics. The modifications included a change in the mounting of the rotating wedge-ring disk to reduce vibration in the optical system and a change in some board level components to increase the thermal stability of the system.

The new system does not appear to have improved temperature stability. A temperature change of 1.4°F was found to cause the distance between the recorded signatures of a fixed image to increase from 4 to 18. In order to reduce the sensitivity of the system to changes in room temperature, a temperature controlled oven has been designed and is being fabricated to control the temperature of the environment around the system.

Preliminary tests have indicated that the noise performance of the system is better than the previous system on a percentage basis. However, its ability to distinguish between different types of defects is no better than the previous system. Personnel from GHC have been contacted and suggestions made to improve the performance of the system through a set of additional modifications. Another system incorporating these modifications should arrive by the middle of April.

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III. PACK INSPECTION, DIGITAL (R. Maher and K. Cox)

A. **Objective**: Develop a full resolution digital inspection system capable of operating at video frame rates.

B. **Results**: Last month, we reported on the development of a new synthetic discriminant function (SDF) which could be encoded on a film and used as a matched spatial filter in an optical correlator. The SDF was designed so that only a single point in the correlation plane need be examined to classify an input image as either good or bad. The numerical value of this point is independent of small shifts in the input image. Because only a single point is needed, the computational effort needed for digital image classification using this SDF is reduced by a factor of approximately 10^5 and can now be carried out in real time. In collaboration with Cary Longest of Engineering, efforts have been made to identify and obtain the hardware needed for the development and testing of a prototype digital "smart camera."

C. **Plans**: Test the concept by computer simulation using the current image library.

IV. INDIVIDUAL CIGARETTE INSPECTION (D. Lowitz)

A. **Objective**: Develop methods for the inspection of individual cigarettes on a making machine.

B. **Results**: An initial survey was made of our current single cigarette inspection capabilities through discussions with personnel in the Engineering Department and in Operations. Several needs were identified including easy to use learning modes in existing inspection systems, a means to monitor cigarette rod/filter abutment integrity, and rejection capability for rod print irregularity.

C. **Plans**: Upgrade our current capability appraisal to include the new Hauni monitors, ORIS and OTIS. Develop an improved analysis system for the CIM 7401 monitor head.

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PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: February, 1988

I. LOW NICOTINE

A. **Objective**: Pilot plant support.

B. **Results**: A total of 29 runs were completed on the one liter laboratory system to help establish operating conditions at the Bermuda Hundred pilot plant. The goal was to determine the minimum AB required to extract to 97% nicotine removal using 200 M/M CO_2.

Results obtained for five component blends are as follows:

Blend	Code	% AB	% Red	Ext Nic %
MT Oriental	J8CP	3.00	92.0	0.07
Thin Bright	J8CL	3.50	97.5	0.07
Bodied Brt	J8CM	4.00	96.0	0.14
Thin Burley	J8CO	1.00	96.2	0.11
Bodied Burl	J8CN	1.50	96.0	0.15

AB % is target AB.

In addition to the above blends, conditions were established for the components of a new ART blend. Results are as follows:

Flue cured	J8HQ	3.80	94.0	0.17
Art Burley	J8HP	1.75	97.0	0.12
Art Blend	J8HO	3.40	97.0	0.09

C. **Plans**: This work is ongoing.

II. LOW NICOTINE

A. **Objective**: Relocate the one liter laboratory to D201.

B. **Results**: Fabrication of the new one liter lab system in D201 is complete. After two shakedown runs, six runs were made on the new system which duplicated runs made in the 7th floor lab to determine if the extractions were comparable. In all cases the D201 system performed as expected. The 7th floor lab has been taken out of service and operations are now conducted in D201.

C. **Plans**: Completed.

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III. LOW NICOTINE

A. **Objective**: Develop fundamental understanding of extraction behavior.

B. **Results**: A correlation for determining AB requirements from initial filler pH and filler titratable acidity was found to hold for the blend components studied to date. The relationship is expressed in milliequivalents of base and hydrogen ion concentration. Presently the AB value obtained from the correlation is for extraction using 200 M/M CO_2.

C. **Plans**: Develop a data base with a range of fillers to better define the correlation for all tobacco types.

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PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: February, 1988

I. OPERATIONS SUPPORT (T. Van Auken, in collaboration with B. Semp)

A. **Objective**: Reduction of alcohol in aftercut formulations.

B. **Results**: Trial runs were conducted with a laboratory scale sonolater at the Sonic plant in Stratford, Connecticut. Stable AC mixtures were prepared with ethanol reductions of 30%, 30% with the alcohol replaced by water, and 100% alcohol removal. The solutions were slightly turbid but stable. Subjective evaluation of the mixtures is underway by Flavor Development.

C. **Plans**: A method will be developed to assess the consistency of AC solutions prepared via sonolator.

II. OPERATIONS SUPPORT (J. Crump, in collaboration with B. Semp)

A. **Objective**: Develop a rapid method of moisture measurement for the stemmeries.

B. **Results**: Good correlation has been obtained between standard OV measurements and a_w as determined by a Novasina Humidat for E45 bright and B34 burley tobaccos equilibrated to three moisture levels as well as several other grades of bright and burley measured on an as is basis. The results to date indicate that the relationship between a_w and OV is not significantly affected by tobacco grade or belt.

C. **Plans**: The range of the measurements will be extended in a more extensive study involving six grades of bright from each of four belts equilibrated to a_w's ranging from 0.4 to 0.8.

III. LOW DENSITY RODS (M. E. Counts, S. Ganeriwala)

A. **Objective**: To compare compression properties of individual low density and control rods and to measure bulk compression responses of control and test filler as a function of moisture and temperature.

B. **Results**: Measured bulk compression properties of filler used in low density and control rods as a function of moisture content. At the standard condition, the stiffness of control filler was different than that of filler with 6% added pectin. However, this difference decreased with an increase in moisture content.

Results of individual cigarette compression, peel tests, and simple stress-strain tests performed on pectin sprayed and control bright lamina were presented to low density rod personnel. A

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Physical Acoustics acoustic emission system was purchased to obtain the bonding information of low density rods.

C. **Plans**: Acoustic emission apparatus will be incorporated into future tests. Further tests will be conducted to verify the above results with pectin. The tests will be expanded to dextran bonded materials.

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PROJECT NUMBER: 1720
PROJECT TITLE: Analytical Microscopy
PROJECT LEADER: V. L. Baliga
PERIOD COVERED: February, 1988

I. STRUCTURE OF LOW SIDESTREAM CIGARETTE PAPERS (Ehrman, Baliga)

A. **Objective**: Examine the ultrastructure of selected cigarette papers in support of the low-sidestream project.

B. **Results**:

High resolution SEM (HR-SEM) photos of large and small sized $CaCO_3$ papers and the 12% $Mg(OH)_2$ P4LP paper were supplied to Barbro Goodman for the Richmond meeting presentation (1,2).

Low sidestream paper containing attapulgite clay was burned and examined using HR-SEM to determine structural changes to clay particles and flax fibers. The rod structure of the clay particle was no longer distinguishable. The gross paper structure did not form a solid sheet, but maintained a disarrayed network. The burned paper network appeared to be coated with a rough glaze (3).

Dr. Donald Sawyer, PM consultant, addressed fundamental questions on the mechanism of reduced sidestream of $Mg(OH)_2$ papers. He suggested that Mg may be forming a metallic salt of cellulose. The salt complex of cellulose may pyrolyse at a lower temperature than the cellulose, and make the formation of a magnesium carbonate intermediate more likely. He recommended doing a gravimetric measurement of $Mg(OH)_2$ that combines with pure flax paper to see if Mg complexed with the cellulose in a stoichiometric relationship (4).

C. **Conclusions**: The clay additive to the paper may create a glazed coating about the flax fiber or the clay may become a structure in itself encasing the cigarette.

D. **Plans**: Previously planned studies on low-sidestream papers will be continued as time permits.

E. **References:**

1. Baliga, V., P.M. Notebook #8412, p. 116.
2. Ehrman, J., P.M. Notebook #8577, p. 42.
3. Ehrman, J., P.M. Notebook #8577, p. 41.
4. Ehrman, J., personal communication with D. Sawyer, Jan., 1988.

II. STRUCTURAL EXAMINATION OF SCE TOBACCO (Sanders)

A. **Objective**: Determine length, width, and area of Bright thin and bodied, Burley thin and bodied and Oriental tobaccos both before and after SCE. Determine structural and elemental changes that may occur during the SCE process.

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B. **Results**: Work has begun with the help of Eddie Thomas to set up a procedure on the Image Analysis unit to determine length, width, and area measurements.

III. RESPONSE TO ANALYTICAL REQUEST (Sanders, Ehrman, Baliga)

A. **Results**:

Two cigarettes, one B&H and one Marlboro, were submitted to analytical microscopy by L. Ryan for photographic documentation of the different holes created by suspected tobacco beetles and roaches. The papers were examined by LM and SEM (1). The Marlboro cigarette had holes with smooth edges and had a tobacco beetle actively eating its way out. The B&H cigarette had holes with edges curled outward and the holes were always in pairs. There was crimping of paper between the paired holes in the B&H cigarette. The B&H holes were sometimes incomplete with paper fibers remaining stretched between the holes whereas the Marlboro holes were complete. Two more paper samples were submitted, one with holes created by roaches and the other with holes created by tobacco beetles. The results obtained using LM and SEM confirmed that the holes created in the Marlboro and B&H cigarettes were, in fact, created by tobacco beetles and roaches respectively (2).

D. Faustini requested a photographic montage of cultured insect larvae using SEM. A method of sample mounting for microscopic investigation was devised (3).

Material collected from fans from the Cabarrus DIET processing plant was examined using LM, SEM, and EDS to determine composition and texture. The major elements present in the material were Ca, Si, C, and O. Other elements such as K, Mg, Ti, Al, P, S, and Fe were also found but differed in concentrations from different areas of the sample. The sample had at least two structurally different regions on the surface. One consisted of a smooth glassy surface while the other appeared rough and brownish in color. The interior structure consisted of enclosed bubbles and was moderately to extremely porous depending on the location examined. The solubility of the material was checked in water, methylene chloride, 100% ethanol, and freon. After each sample soaked overnight in its respective solvent at room temperature, the sample in water was only partially soluble, and the water had a murky brown discoloration. The sample in methylene chloride was partially soluble and the solvent was dark brown in color. The same was true for the sample in ethanol. The sample in freon was insoluble and the freon had no discoloration. The material continued to float in the freon solution. The crushed sample was not paramagnetic and did not burn readily (4).

B. **Plans**: Work on the photographic montage will continue.

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C. **References**:

1. Sanders, K., P.M. Notebook #8374, pp. 177-186.
2. Sanders, K., P.M. Notebook #8374, pp. 187-192.
3. Sanders, K., P.M. Notebook #8374, p. 193.
4. Ehrman, J. and Baliga, V., 'Analysis of 'buildup' material from Cabarrus DIET process', memo to D. Watson, February 22, 1988.

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Source: https://www.industrydocuments.ucsf.edu/docs/xnxm0000

PROJECT NUMBER: 1730
PROJECT TITLE: Plant Cell & Tissue Culture Research
PROJECT LEADER: I. L. Uydess
PERIOD COVERED: February, 1988

I. TOBACCO IDENTICAL PRESERVATIVES

A. **Objective**: To develop procedures and to establish microbiological screens for the evaluation of new, nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. **Status & Results**:

1. Antimicrobial/Preservative Screens:

a. Phase I Screens: Several aqueous phase fractions of oriental tobacco (grade LSB) and one sucrose ester fraction from samsun tobacco (D. Danehower, N.C.State University) were tested in a scaled-down version of the agar inclusion assay. The aqueous fractions were found to have marginal activity at about 5000 µg/ml, while the sucrose ester fraction (92% pure) was observed to have substantial activity at or below 500 µg/ml. A similar result to this was reported previously for a less-well purified sample of sucrose esters isolated from oriental tobacco (grade LSB, D. Teng, see January, 1988 monthly).

2. Microbiological Investigations:

a. Phase II Screen: Additional samples of sterile, Park 500 SEL have been induced to spoil (increase in pH, cell number, and odor) under controlled laboratory conditions in support of the development of a standard operating procedure for a Phase II screen. In each case, fresh samples of autoclaved SEL were inoculated with the mixed culture of bacteria obtained from a previously spoiled sample of SEL. After multiple serial passages, four gram-positive bacteria continued to comprise the mixed culture, although the relative ratios of each has been observed to vary with time and increasing pH as spoilage of the SEL progressed. Two of the organisms appeared to predominate at more acidic pHs (pH 5.0 - 6.5), while the other two predominated at the more neutral and basic pHs (pH 7.0 - 8.5). Similar attempts to employ each of the isolates alone have proven unsuccessful. In each case, the individual isolates have not been able to grow successfully in sterile SEL (three die off, one grows marginally).

A preliminary Phase II control screen was successfully run in sterile SEL using the mixed culture exposed to various levels of propylparaben in PG. After 48 hrs, the cell counts (CFUs) in the samples exposed to 300, 500 and 1000 µg/ml PP-PG were found to be 3.5 logs lower than that of the

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control flasks (approximately, 1.0 E4 vs 5.0 E7, respectively). In addition, the relative sensitivity of each of these isolates to PP-PG was evaluated in the Phase I, agar-inclusion assay. Two of the organisms were found to be less sensitive to PP-PG than the others in this assay system.

b. Biochemical Investigations: Chemical evaluations of the chemistry of spoilage are also being conducted in collaboration with members of the Analytical Research Division. Recently, ^{13}C-NMR (Dr. Jan Wooten) has proven to be very effective in following the time course of chemical change in samples of laboratory spoiled SEL. This information will be used to help establish biochemical criteria for the spoilage of SEL in support of the overall goals of this program.

c. Adhesive Spoilage Study: Samples of a starch-based adhesive have been received from B. Semp for the investigation of spoilage. Multiple aliquots of this material are being prepared so that they can be exposed to the air to promote contamination.

C. **Conclusions**:

1. Sucrose esters continue to exhibit substantial antimicrobial activity in the Phase I screens.

2. Laboratory induced spoilage in SEL appears to be the product of the metabolic activity of one or more gram-positive bacteria which may have complementary (or interdependent) metabolic requirements. Additionally, the progression of spoilage is paralleled by a characteristic shift in the relative composition of the mixed culture as the pH of the SEL rises.

D. **Plans (March, 1988)**:

1. Additional samples of the sucrose ester fractions from both samsun and LSB oriental tobaccos will be tested in the Phase I, agar inclusion screen.

2. Further analysis of the biochemical and microbiological characteristics of spoilage will be conducted in support of the development of a Phase II, SEL preservative screen.

3. The spoilage of a starch-based adhesive (provided by B. Semp) will be investigated. Propylparaben in PG will be evaluated as a possible preservative for this material.

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PROJECT NUMBER: 1752
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke
PROJECT LEADER: J. O. Lephardt
WRITTEN BY: M. E. Parrish
PERIOD COVERED: February, 1988

I. CIGARETTE SMOKE INVESTIGATIONS

A. **Objective**: Develop a method for the analysis of aldehydes in mainstream(MS) and sidestream(SS) cigarette smoke using 2,4 DNPH derivatization followed by separation and detection using an HPLC procedure.

B. **Results**: The trapping and reaction conditions have been optimized. SS will be collected for 2 seconds at 5,20,35, and 50 seconds using one cigarette at 5 seconds, a second cigarette at 20 seconds, etc. for a total of four cigarettes per SS trap. MS will be collected from four cigarettes for each MS trap. Samples from the traps will be injected onto the HPLC for separation and quantitation of the aldehydes using appropriate 2,4 DNPH standards.

C. **Conclusions**: The method for the SS analysis of selected aldehydes has been developed and easily can be applied to MS.

D. **Plans**: Data will be collected to assess method reproducibility and comparisons will be made with data from other analytical techniques.

E. **References**:

E. Thomas, P.M. Notebook #8502, pp. 72-74.

A. **Objective**: Develop a method for the real-time measurement of acrolein in MS and SS smoke using tunable diode laser(TDL) spectroscopy.

B. **Results**: Experiments were performed and completed which showed that heated inlet lines were not necessary. The potential interference of relatively high concentrations of methanol was evaluated and conditions optimized to eliminate this effect. Using the acrolein permeation calibration of 57.5 ug/min., the SS delivery for Monitor 24 cigarettes (n=34) was 560+/-50ug/cigt (70+/-7ug/min.), and 0.84+/-0.07 mg/g of tobacco and paper consumed.

C. **Conclusions**: The TDL method has been developed for MS and SS acrolein analyses(although not simultaneously) and SS data have been collected for the six circumference model cigarettes.

D. **Plans**: MS acrolein deliveries will be determined for these selected models and provisions will begin for reactivating the MS and SS formaldehyde methods on the second TDL system.

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E. **References**:

M. Parrish, P.M. Notebook #8548, p. 90.

A. **Objective**: Develop a method for the simultaneous determination of total ammonia in the MS and SS whole smoke for a single cigarette using ion chromatography (IC).

B. **Results**: Several experiments were performed to determine the distribution of total ammonia in the SS particulate and gaseous phases using differently designed impaction traps. Less than 2% of the total ammonia was detected in the SS TPM collected in the impaction traps and 98% was found in the gas phase. The established method was applied to the six circumference model cigarettes and the results confirmed the observations obtained from using the gaseous ammonia TDL procedure.

C. **Conclusions**: Sufficient data have been collected on a number of different cigarette samples to conclude that the total ammonia deliveries of SS whole smoke generally exceed by 10 to 20% the values obtained for SS gaseous ammonia using the TDL.

D. **Plans**: The MS/SS total ammonia whole smoke method using IC is now the method that will be used as needed. Priority will shift to developing methodology for quantitating benzene and toluene in MS and SS smoke.

E. **References**:

1. H. Randolph, P.M. Notebook #8475, p. 102.

2. Randolph, H., "The Simultaneous Determination of the Total Ammonia Delivery in MS and SS Smoke for Cigarette Models with 17, 20, and 24.8 mm Circumference," memo to J. Lephardt, February 3, 1988.

II. ANALYTICAL SUPPORT TO R&D AND OTHER P.M. DEPARTMENTS

A. **Objective**: Perform pyrolysis-GC/MS analyses as requested.

B. **Results**:

A sample of Angstron Tech. Compound #5 was examined at 315 and 590°C. Six compounds were identified at 590°C.

The volatile pyrolysis products of a cigarette paper sample P8AB were compared to two control paper samples with no compounds observed in the P8AB sample that were not also observed in at least one of the control papers.

C. **References**:

1. Magin, D., "Pyrolysis-GC/MS of Angstron Tech. Compound #5 U.V. Fluorescing Material," memo to R. Cox, February 5, 1988.

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2. Magin, D., "Curie-Point Pyrolysis-GC/MS of Cigarette Paper P8AB," memo to R. Cox, February 11, 1988.

A. **Objective**: Perform GC/MS analyses as required.

B. **Results**:

A sample of a reaction mixture (#8177-199) from D. William was evaluated and it was determined that the expected compound was a minor component of the mixture.

C. **Plans**: Continue to provide pyrolysis-GC/MS and GC/MS support as needed and to initiate a systematic study of tobacco pectin.

A. **Objective**: To perform infrared qualitative analyses of commercial products prior to use in P.M. facilities as part of the Material Evaluation Program.

B. **Results**:

Samples of tube belts were identified as a combination of natural cellulose and polyethylene terephthalate fiber. Samples of conveyor belts were determined to have polyisoprene and polyester urethane.

Samples of machine parts submitted included a plastic cover of polyvinyl chloride, a white rubber sleeve of methyl silicone rubber, and a machine label of methyl/methacrylate.

C. **Plans**: Continue the analyses required in support of the Material Evaluation program.

D. **References**:

M. Griff, P.M. Notebook # 8554, pp. 53-59, 61-63.

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PROJECT NUMBER: 1754
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components
PROJECT LEADER: J. Wooten
PERIOD COVERED: February, 1988

I. SOLID STATE NMR

A. **Objective**: Identify lignin bonding patterns in tobacco using ^{13}C CP/MAS NMR (J. Wooten).

B. **Results**: Last month we reported that ^{13}C CP/MAS NMR failed to show incorporation of ^{13}C into L. Leucocephala plants fed β-^{13}C ferulic acid. In a new sample preparation prepared by Dr. Norman Lewis' group at Virginia Tech, high levels of ^{13}C incorporation were observed. The results are very interesting because the signals from the different enrichment sites resemble the signal pattern in synthetic ("DHP") lignin prepared from [β-^{13}C]coniferyl alcohol. This is in contrast to wheat plants that indicated a bonding pattern distinctively different from the synthetic lignin. It appears that the synthetic material is a good model from lignin in some cases but not in others.

C. **Plans**: Similar experiments are in progress with tobacco.

II. SOLUTION NMR

A. **Objective**: Determine the solution conformation of 6-alkyl cyclohexen-1-one derivatives (R. Bassfield, K. Podraza).

B. **Results**: ^{1}H and ^{13}C spectral assignments were made on a series of compounds using both 1D and 2D techniques. The preferred solution conformation was determined to have the 6-alkyl group in the equatorial position. This conformation was observed not to be conserved when the cyclohexenone is reduced to the di-alcohol, rather a mixture of both conformers were detected in the NMR spectra.

A. **Objective**: Use NMR to characterize different pectins used as foamed binders (R. Bassfield, S. Baldwin).

B. **Results**: GPC columns have been set up to separate low molecular weight fractions of enzymatically degraded pectin.

C. **Plans**: Isolation of the pectin fractions will begin soon. NMR characterization of the individual fractions will be performed.

A. **Objective**: Determine the enantiomeric purity of nicotine isolated from smoke condensate (R. Bassfield, T. Izac).

B. **Results**: The ^{1}H NMR of the condensate isolated nicotine was monitored after successive additions of an optically active shift reagent. Separation of the enantiomers was achieved after a mole

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ratio of 0.45 of reagent was added. Because of severe signal broadening from the shift reagent, an accurate assesment of the optical purity may require deconvolution of the overlapping H-2′ resonances.

C. **Plans**: Different shift reagents will be used to try to obtain better separation of the H-2′ signals.

A. **Objective**: Monitor the effects of SEL spoilage by ^{13}C NMR (J. Wooten, I. Uydess)

B. **Results**: SEL solutions were examined by ^{13}C NMR. Fresh SEL contained signals primarily from glucose, fructose, and nicotine in addition to signals from some unidentified materials. After spoilage, a strong signal from acetate appears and an unidentified signal at 36 ppm appear while the sugar signal intensity is greatly reduced. As spoilage progresses, the sugar signals as well as all other signals including the acetate signal disappear except nicotine. It was found that the signal position of the C-3 of nicotine can be conveniently used to monitor the pH of the solution. A strong bicarbonate peak appears in the highly spoiled SEL.

C. **Conclusions**: NMR can conveniently be used to monitor fructose, glucose, nicotine, acetate, and a few other components in SEL.

III. ANALYTICAL ELECTRONICS

A. **Objective**: Modify the portable air sampling system (PASS) for improved performance and reliability (S. Marrs, G. Baker).

B. **Results**: The present PASS system allows the CO pump to be calibrated for a flow rate of 500 ml per minute. The calibration adjustment range does not permit calibration at lower flow rates. A new pump was tested to evaluate the power requirements and response characteristics for a flow rate of 200 ml per minute. Three regulator power regulator circuits were designed in an attempt to provide the pump with constant low voltage power supply needed for long term operation.

C. **Plans**: PASS design modifications are still in progress.

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PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: C. S. Kroustalis
PERIOD COVERED: February, 1988

ANALYTICAL FLAVOR SPECIFICATIONS

A. **Objective**: To develop analytical and sensory specifications for incoming materials and flavors used at the Flavor Center and other QA facilities.

B. **Results**: All of the German flavors received have been evaluated. Some fomulated flavors will require additional determinations for solvent(s) content before final specifications can be written. We are currently writing specifications for the remaining materials and we anticipate completion of all specifications by mid-March. We have also been collaborating with Flavor Center personnel on the original set of materials which they began evaluating.

C. **Plans**: Complete the solvent determinations for the remaining compounded German flavors and write specifications for all flavors we have evaluated. Review all specifications for accuracy prior to sending them to the Flavor Center.

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PROJECT NUMBER: 1758
PROJECT TITLE: Tobacco Cell Wall Research
PROJECT LEADER: G. H. Bokelman
PERIOD COVERED: February, 1988

I. BLEND COMPOSITION ANALYSIS (G. Bokelman, J. Stimler and General Analytical)

A. **Objectives**: (1) Compare the blend compositions of Barclay Lights Ultra Thins, Regular and Menthol, with those of Capri, Regular and Menthol, (2) predict the blend composition of Silkroad (Japan) and (3) predict the blend compositions of six brands of Korean cigarettes.

B. **Status**: Analytical data for the above mentioned cigarettes were processed by our present mathematical model for determining components in a tobacco blend. In this model, four chemical predictors (total ash; aspartic acid; β-methylvaleric acid, ratio of sample to standard; and cellulose index) were used as independent variables for the four dependent variables: bright lamina, burley lamina, Oriental leaf, and combined bright and burley stems. Stems, of course, may be present in a number of forms, including expanded stems and reconstituted tobaccos.

There are important **caveats** regarding the application of this methodology. This method of predicting blend components should give accurate results when the actual tobacco components to be used are incorporated into the database, a condition which for practical purposes only can be met for products manufactured by Philip Morris. Inaccurate results may be obtained if this method is applied to unknown blends or blends whose components differ markedly in chemistry from those used in the database. The database for this study consisted of typical commercial tobacco components used by Philip Morris for domestic cigarette production in the first quarter of 1987. We have not yet generated either a "Japanese database" or a "Korean database."

C. **Results**: The predicted blend compositions for the respective versions of Barclay Lights Ultra Thins and Capri are very similar. Perhaps the most interesting findings are the changes in the two menthol versions compared to the two regular versions (increased content of bright lamina, decreased contents of burley lamina and Oriental leaf). The similarity between the respective versions of Barclay Lights Ultra Thins and Capri is supported by C.I. data for contents of total alkaloids, total reducing sugars, reconstituted tobacco, expanded stems and expanded tobacco. For the sake of comparison, the predicted blend composition of Barclay also has been included.

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Table 1. Predicted Blend Compositions for B&W Cigarettes
(values expressed as percentage of total)

Component	Barclay	Barclay Lights Ultra Thins Regular	Capri Regular	Barclay Lights Ultra Thins Menthol	Capri Menthol
Bright Lamina	38	40	42	49	57
Burley Lamina	29	31	26	22	16
Oriental Leaf	18	20	22	14	17
Stem	15	9	9	15	10

Previously we made relative comparisons among the blend compositions of Marlboro Lights (Japan) and four cigarette brands produced by Japan Tobacco Inc.--Libera Milds, Mild Seven, Mild Seven Lights and Caster (1). Compared to all these other brands, Silkroad (Japan) is notable for its extremely high content of bright lamina and its low contents of stem and burley lamina (see Table 2).

The predicted blend compositions for the six brands of Korean cigarettes are listed in Table 3. Significant variations among the predicted blend compositions are evident (2). Two of the brands are notable for their absence of Oriental leaf.

We believe that the predicted blend composition values listed in Tables 2 and 3 should be useful for making **relative comparisons**. However, keeping in mind the caveats listed previously, the accuracy of individual values must be considered somewhat suspect.

Table 2. Predicted Blend Compositions for Japanese Cigarettes
(values expressed as percentage of total)

Component	Mild Seven	Mild Seven Lights	Caster	Silkroad	Libera Milds
Bright Lamina	48	44	48	70	41
Burley Lamina	20	23	12	9	33
Oriental Leaf	6	5	4	7	8
Stem	26	29	36	14	18

Table 3. Predicted Blend Compositions for Korean Cigarettes
(values expressed as percentage of total)

Component	Pine Tree Golden Lights	"Orange" (See **)	Cheong Ja	Silver Reed	Eunhasu	Eighty Eight
Bright Lamina	50	41	49	59	50	43
Burley Lamina	19	24	21	13	23	22
Oriental Leaf	6	0	0	6	2	6
Stem	25	35	31	22	25	29

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** "Orange" is the name we are using to designate a brand whose actual name is written only in Korean characters. The cigarette pack for this brand has a distinctive orange wrapper.

D. **Plans**: Islands, a new cigarette brand produced by RJR Nabisco for sale in Japan, has been submitted for chemical analyses. As soon as these analytical values are available, we will estimate its blend composition.

E. **References**:

1. Bokelman, G. H. and J. O. Stimler, memo to C. L. Irving, "Blend Composition of Libera Milds (Japan)," December 3, 1987.

2. Bokelman, G. H. and J. O. Stimler, memo to C. L. Irving, "Blend Compositions of Silkroad (Japan) and Six Korean Cigarette Brands," February 2, 1988.

II. CHEMICAL ANALYSIS OF UNKNOWN FLOCCULATED MATERIAL (S. Baldwin)

A. **Objective**: Characterize an unknown material submitted for analysis.

B. **Background**: An unknown fluffy, gray-brown solid material was found in the tobacco hopper, following an attempt to prepare low density rod cigarettes (from an all-lamina blend of uncased tobacco) using dextran as a binder. The unknown material, suspected of being dextran, was referred to as "floc."

C. **Results**: After conducting a variety of solubility tests and specific carbohydrate analyses, it was concluded that the unknown material was indeed dextran (1). Its dark color may have been acquired from tobacco dust or some type of oil.

D. **Plans**: No additional work in this area is planned.

E. **Reference**:

1. Baldwin, S., memo to V. Manual, "Chemical Analysis of an Unknown Flocculated Material," February 19, 1988.

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PROJECT NUMBER: 1902
PROJECT TITLE: Tobacco Microbiology
PROJECT LEADER: D. J. Ayers
WRITTEN BY: D. K. Chadick
PERIOD COVERED: February, 1988

I. ALTERNATE HUMECTANT PROGRAM

A. **Objective**: To determine the effects of storage time and temperature on Pilot Plant produced RL containing Isosweet (2 or 4%) as a replacement for the glycerol component.

B. **Status**: The study was completed and a memo was issued (1).

C. **Plans**: None at this time.

D. **References**:

1. Crockett E. A., Microbial Analyses of RL-TC and RL-150B With and Without Isosweet and K-pp in Place of PG and G as Part of the Alternate Humectant Program (AHP). Memo to E. Mooz; 1988 February 11.

II. HOGSHEAD, BOX, AND BALE STUDY

A. **Objective**: Microbially analyze Bright tobacco from the 1987 crop for baseline data and the 1986 Crop of bright after 1 year of warehouse storage as part of the Hogshead, Box and Bale 3 year study.

B. **Status**: Baseline microbial counts were established for Bright from the 1987 crop year (1). The microbial counts from bright tobacco after one year of storage were completed and indicated no significant change in counts between the samples stored in hogsheads, boxes or bales (2).

C. **Plans**: Microbial counts will be obtained after 1, 2 and 3 years of storage.

D. **References**:

1. Crockett E. A., Microbial Analyses of Bright Tobacco as Part of the Hogshead, Box, and Bale Study (initial counts from the 1987 crop). Memo to H. Webb; 1988 January 15.

2. Crockett E. A., Microbial Analyses of Bright Tobacco as Part of the Hogshead, Box, and Bale Study (1986 crop year after 1 year of storage). Memo to H. Webb; 1988 January 18.

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III. MICROBIAL ANALYSES OF IRRADIATED CIGARETTES

A. **Objective**: To microbially analyze typical blended cigarettes (with and without aftercut casing) that had been packaged and then gamma-irradiated with 0, 0.50, 0.75, 1.0, or 2.0 millirad(s). This microbial investigation is part of the Taste, Odor, and Stale program.

B. **Status**: Work for this study (1) was completed and a memo was issued (2).

C. **Plans**: None at this time.

D. **References**:

1. Gaines, O. M. 1902 Monthly Progress Report Acc. #88-010; January, 1988.

2. Gaines O. M. Microbial Analyses of Irradiated Marlboro blend Cigarettes (with and without aftercut casing). Memo to D. B. Spruill; 1988 February 17.

IV. SHREDDED STEM STUDY (FOR PROJECT ART)

A. **Objective**: To determine if the ART process changes the microbial numbers in shredded bright stems (12% and 35% OV's) after being stored for 0, 24, 48, 72 and 96 hours and 1, 2, 4, 8, 12 weeks at 37°C.

B. **Results**: Shredded bright stem samples (12% and 35%) were tested before the citrate spray, after the citrate spray, immediately post-ART (35% OV only), and after drying. In this first experiment there were no significant increases seen from any of the samples after 4 weeks of storage (1).

C. **Plans**: This is an on-going study.

D. **References**:

Jones, J. Notebook No. 8590.

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PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Physiology and Biochemistry
PROJECT LEADER: H. Y. Nakatani
PERIOD COVERED: February, 1988

I. LOW NICOTINE STUDY

A. **Objective**: To investigate the biochemistry of the nicotine biosynthetic pathway at putrescine N-methyltransferase (PMT) and specifically to isolate PMT from tobacco root extracts.

B. **Status**: The harvest of the roots from the group nine plants was staggered to examine the yield post-topping since the root mass from the last two groups was quite small. The extraction of roots from group nine, one week (PM53) and two weeks (PM54) post-topping was completed to the 40-65 % ammonium sulfate stage and treated to allow application to the first equilibrated phenyl-Sepharose column (1).

Another octyl-Sepharose column was investigated using a NaCl rather than ammonium sulfate gradient. This column did not enhance the resolution over the phenyl-Sepharose columns currently in use (3,4). Hydroxyapatite chromatography was also examined further. Binding characteristics were determined; however, substantial loss in PMT activity will likely preclude the use of this support medium in the purification scheme (2).

Multiple PMT-active samples were run on the 1 X 40 cm Sephadex G-100 column (minus glycerol) to obtain material for the Hazleton antibody contract. Pooled fractions were concentrated to approximately 22 ug/150 ul (total 5.8 ml) (6) which had a specific activity of 7.3×10^4 units per mg protein (3). [One unit equals 1 nmole of SAM (^{14}C-S-adenosylmethionine) 30 min of reaction time.] The ammonium sulfate fraction had a specific activity of 1.8×10^3 units per mg protein (3).

A commercially prepacked Sephacryl S200HR (Pharmacia) gel filtration column (1 liter bed volume) was set-up and is being equilibrated and calibrated for use (6). The PMT-active fractions were pooled from the first phenyl-Sepharose column and precipitated with ammonium sulfate (65 % saturation) to be applied to this column.

Several ELISA (enzyme linked immunosorbent assay) experiments, using a rabbit antibody against phenylethanolamine N- methyltransferase (PNMT-Ab)) were conducted to compare the color reaction (alkaline phosphatase) with PMT and PNMT samples (total protein concentration from 1-4 ug). A twenty to forty-fold enhancement of the color reaction was observed with the authentic PNMT and the PNMT-Ab over the PMT samples and the generic antibody (7). A miniblotting device to determine the cross-reaction of antibody(ies) against proteins which have been transferred to nitrocellulose and PVDF (polyvinylidene difluoride) paper (Western blot) is also being examined (7). Preliminary background absorption problems are being addressed. This device will allow use of

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approximately 50-75 ul of antibody solution versus many milliliters for the classical methods, a concern especially with monoclonal antibody production. A magnetic bead (MagniSort bead, DuPont) bound with an antibody against rabbit IgG is also being examined and shows promise for our screening efforts (7).

The technique of photoaffinity labeling of PMT with ^{3}H-S-adenosyl-methionine (SAM), using UV irradiation is currently under investigation (8). Our initial examination has been conducted with PNMT. The conditions for incorporation appear to be satisfactory and PMT samples will be exposed to a similar treatment. Controls were run with either excess SAM or S-adenosylhomocysteine. ^{3}H-S-adenosyl-methionine assays have been conducted as necessary to support the PMT purification effort (3).

C. **Plans**: The next group of hydroponically-grown tobacco plants (group 10) will be topped sometime near mid-March. The roots will be harvested thereafter and extracts will be prepared. The purification of PMT will continue. Studies will continue with various chromatographic media. Sequential samples will be applied to the gel filtration column (1x40 cm). The large-scale, commercially prepacked, Sephacryl gel filtration column will also be tested with PMT containing fractions from the initial phenyl-Sepharose column. Antibody binding studies will continue using MagniSort-beads, Western blots and microtiter plates. ^{3}H-SAM labeled PNMT and PMT samples will be examined by SDS-PAGE.

D. **References**:

1. Sherwood, K. R. Notebook No. 8545.
2. Malik, V. S. Notebook No. 8542.
3. Nakatani, H. Y. Notebook No. 8384
4. Dunn, R. L. Notebook No. 7899.
5. Sykes, A. Notebook No. 8526.
6. Yu, T. Notebook No. 8381.
7. Mooz, E. D. Notebook No. 8599.
8. Davies, B. D. Notebook No. 8005.

II. ALTERNATE HUMECTANTS (PG/G-FREE SHEETS/CIGARETTES) (1)

A. **Objective**: To find a plasticizer/humectant system that provides acceptable sheet materials without glycols (PG/G).

B. **Results**: Subjective evaluation was made of the 100% cigarettes from production plant RL and RCB sheets containing K-pp in place of PG-pp. No significant difference was found between the test and the control cigarettes. Subjective evaluation was made of the 100% cigarettes made from PG/G-free RCB handmade sheets produced in the Pilot Plant. A level of 4% isosweet substituted for PG/G gave the best results.

C. **Plans**: Memos will be issued by Flavor Development summarizing the data from the subjective evaluations described above. Subjective evaluation will be made of the 100% cigarettes from PG/G-free RL

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sheets made in the Pilot Plant. Plans will be made to produce PG/G-free RL and RCB feedstocks at the production plants in March.

D. **References**:

1. Mooz, E. D. Notebook No. 8599.

III. ALTERNATE HUMECTANTS (GLYCERINE-FREE SHEET/CIGARETTES) (1)

A. **Objective**: To provide acceptable glycerine-free (G-free) RLTC, RL150B, and RCB sheets and G-free cigarettes for the European market.

B. **Results**: Subjective evaluation was made on the 100% cigarettes from production plant RL and RCB G-free sheets. The RL products were found to be similar, but the RCB models were considered "atypical". A memo was issued summarizing the results (2). As a result of these subjective tests, G-free RCB was remade at the BL plant. G-free, cased ET and ESB were also made.

C. **Plans**: A POL test is planned with a G-free Marlboro type cigarette and a Marlboro in which the recons will be made with no class tobacco to be tested versus a control Marlboro cigarette.

D. **References**:

1. Mooz, E. D. Notebook No. 8599.
2. Spielberg, H. L. Reconstituted Tobacco without Glycerine. Memo to Mr. D. Knudson; 1988 February 2.

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PROJECT NUMBER: 2106
PROJECT TITLE: Cigarette Performance and Design
PROJECT LEADER: R. W. Dwyer
PERIOD COVERED: February, 1988

I. FILTER VENTILATION (D. Simpson)

A. Objective: Develop a predictive relationship between filter ventilation and cigarette construction.

B. Status: A mathematical model relating filter ventilation levels with tipping-paper blue-clamp pressure drops and plug-wrap CORESTA permeabilities is being developed. Experiments have been designed in which a wide variety of tipping-paper/plug-wrap combinations will be used to construct cigarettes. Vendors have provided us with companion bobbins of both materials, each from the same slitter set.

II. CIGARETTE FILTER EFFICIENCY VARIABILITIES (L. Goodwin)

A. Objective: Determine the variability in efficiencies of filters from a production run of cigarettes.

B. Status: A case of Marlboro Lights 85's has been obtained for testing. A random sampling of filters from these cigarettes are being evaluated with our filter efficiency test station. This instrument allows us to measure efficiencies as functions of flow rate and aerosol particle size.

III. CIGARETTE DELIVERIES (J. Kao)

A. Objective: Develop a predictive model of cigarette TPM, nicotine, and water deliveries as functions of cigarette-design parameters.

B. Status: CTS has provided us with puff-by-puff deliveries as functions of puff volumes for a series of unfiltered cigarettes. A model is being developed for such data based on the generation and filtration of the smoke aerosol.

IV. COMPUTER AIDED CIGARETTE DESIGN (D. Leister and L. Watt)

A. Objective: Develop a computer-based system for predicting the effects of cigarette design on cigarette performance.

B. Status: The appropriate hardware and software for this program have been received and installed. Two routines have been developed on this system, one which calculates capability curves for any CA tow item, and one which calculates tows for meeting specified pressure-drop and firmness targets. Currently, routines are being developed for predicting puff-by-puff TPM deliveries as functions of cigarette design.

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PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: February, 1988

I. FLAVOR/ODOR CHEMISTRY (Paine, Secor, Seeman)

A. **Objective**: To develop new technologies for smoke deliveries of desired flavorants; to prepare new substances for flavor/odor evaluation; to develop methodologies for the analysis of subjective data; to derive relationships between physicochemical parameters and subjectives.

B. **Results and Plans**: Because of the continued interest in pyrazine release agents in general and in tetramethylpyrazine (TMP) release in particular, we have begun and made excellent progress in preparing a series of TMP:polyalcohol release agents. The polyalcohols are meant to include sugar derivatives or sugar analogues. Several of the molecules prepared to date have been pyrolyzed by D. Magin using the pyroprobe; release of TMP occurred in all examples constructed for that purpose (i.e., β-hydroxyethylpyrazines). No TMP was observed from the pyrolysis of TMP-release agent precursors.

Three trimethylpyridine release agents were prepared, all substituted at the 2-position. The rates of trimethylpyridine release will be compared with the analogous 4-substituted pyridines which have bulky tert-butyl groups at C_2 and C_6 and thus cannot participate in bimolecular elimination pathways.

Pursuant to a request from Jośe Nepomuceno and Roger Comes, 4L of a 56% (w/w) solution of glucose menthyl carbonate in propylene glycol was prepared and delivered.

A paper on the odor profiling and multidimensional scaling of various alkyl benzenes and alkyl pyridines is nearly completed. This involves the separation of multidimensional percepts of odor quality of these aromatic compounds into specific odor factors and the subsequent correlation of these particular odor factors with chemical descriptors.

II. CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS (Secor, Seeman)

A. **Objective**: To obtain structural information about important tobacco constituents/flavorants; to develop information on cluster formation and chemical reactions in clusters.

B. **Results and Plans**: Studies have continued with E. Bernstein (Colorado State University) on substituted benzenes, pyridines and pyrazines. Additional styrenes, anetholes, and anisoles were prepared and sent to CSU for laser jet studies. Reports on the alkyl and alkoxy aromatics have been completed and submitted to the Manuscript Review Board, and reports on allyl and vinyl

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aromatics are being written and/or modified. Plans have been made for the next year of collaborative research, and a proposal has been completed. We have also completed an intensive and productive visit to CSU.

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PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
PROJECT LEADER: R. A. Comes
PERIOD COVERED: February, 1988

I. SIDESTREAM SMOKE

A. **Objective**: Conduct analyses on mainstream and sidestream smoke to determine qualitative and quantitative differences between control and reduced visibility cigarettes. Develop methods to analyze for selected components. Demonstrate effective methods for reduction or removal of these components.

B. **Status**: 1. Sidestream gas phase analysis is now being routinely carried out with a 60 M, 1 micron film thickness DB-5 column. Sidestream gas phase from cigarettes prepared with 35% $Mg(OH)_2$ and 35% $CaCO_3$ wrappers has been compared with that from Marlboro Lights as control. The effluent was monitored by FID and NPD and the data examined using the PE LIMS system. Gas phase composition was studied by MS by direct syringe injection of 50 ml samples.

2. Several different samples of sidestream smoke have been analyzed using the silica-gel trapping technique.
   a. The sidestream smoke from a cigar was collected and worked up in the usual fashion.
   b. Sidestream from Marlboro Lights was collected under both static and dynamic (puffing) conditions.
   c. Samples of the 35% $Mg(OH)_2$ and 35% $CaCO_3$ wrapped cigarettes were studied vs Marlboro Lights as control.
   d. RL baseweb samples prepared individually from bright, burley and oriental were obtained and smoked. An LTF model was also smoked in this series. These essentially "cellulose only fillers" were chosen to compare the ethyl acetate fractions with those of "normal" blends.

C. **Results**: 1. Good reproducibility is obtained for the gas phase analyses except for the early, low molecular weight compounds which are trapped unreproducibly in the liquid-nitrogen cooled trap at the head of the analytical column. The samples examined produced very similar profiles with both NPD and FID detectors. The gas phase from Parliament Lights 100's was studied for compound identification. Fifty-five components ranging from carbon dioxide to limonine have been determined to date. These identifications will be useful for establishing elution order on non-polar columns and for comparison with whole smoke work carried out with the Finnegan MS.

2. A few new compounds have been identified by MSD. The list of compounds identified to date has been consolidated, revised and additional and corrected CAS numbers added.
   a. No significant differences were seen between cigar smoke, control cigarette smoke and low ss cigarette smoke.
   b. No significant differences were observed when ss smoke was collected from statically or dynamically burned models.

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c. Small quantitative differences were observed between the low sidestream models utilizing different wrappers.
d. The FID profiles of the baseweb samples vs normal blends were surprisingly similar. However, examination by MSD revealed significant qualitative differences. The baseweb samples were very similar to each other but appear to contain more cellulosic degradation products than do the normal blend samples.

D. **Conclusions**: 1. Significant progress has been made in improving gas phase chromatographic resolution. However, as seen previously, few differences are seen between the gas phase profiles of various cigarette types.

2. Few differences have been observed in the sidestream smoke between the various models studied utilizing the silica gel collection technique. However, striking differences have been observed between the ethyl acetate fractions of the sidestream and mainstream smoke from the same sample with this method. This indicates that the method is capable of discerning differences if they are there to be seen and that the similarity of sidestream samples is simply a reflection of the true nature of the samples. That is, the chemical composition of sidestream or mainstream smokes from different tobaccos is in fact very similar.

E. **Plans**: Continue GC/MS studies on the mainstream and sidestream smoke from cigarettes smoked under both static and dynamic conditions. Generate data from multiple runs to determine qualitative and quantitative mainstream and sidestream gas phase comparisons.

II. Miscellaneous

A. **Objective**: 1. Upgrade computer capabilities for the Project and for the Division as required.

2. Conduct sidestream smoke visibility measurements as required.

3. Determine experimental conditions necessary to support the program to incorporate selected ingredients into cigarette models to improve sidestream smoke properties.

4. Conduct GC/MS and pyrolysis/GC/MS analyses as required.

B. **Results**: 1. The upgrade to the P.E. system has been delivered, installed and is operational. A file transfer protocol is available to allow direct transfer from the Perkin-Elmer system to the UNIX system. The 850Mb disk drive has been installed and is currently undergoing testing.

2. Several sets of cigarettes have been studied for visibility and related data. These include those to determine the effect of $CaCO_3$ wrappers as compared to the $Mg(OH)_2$ models.

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3. Procedures are being studied to conduct these analyses utilizing GC and GC/MS.

4. Several requested analyses were carried out.

C. **Conclusions**: 1. Greatly expanded file transfer and data manipulation capability is now possible through the upgraded systems.

2. Some differences exist between the visibilities determined for $CaCO_3$ wrapped cigarettes when compared to $Mg(OH)_2$ models. The new small particle size $CaCO_3$ papers appear to give less reduction in visible sidestream smoke but apparantly have positive subjective properties when compared to the $Mg(OH)_2$ models of comparable composition.

3. Methods will shortly be available to quantitatively determine the detected amounts of vanillin found in the MS TPM, SS TPM, SS chamber wash and butt from cigarettes to which known amounts of vanillin have been applied.

4. Vanillin was the only detected component found in the 300 deg C pyrolysis of CR-2673 (glucovanillin). At 600 deg, vanillin (63%), catechol (27%), unknown m/z 150 (6%) and hydroxybenzaldehyde (3%) were detected. No impurities were found in the 300 and 600 deg C pyrolyses of Darco-G charcoal.

D. **Plans**: 1. Continue upgrades to the current systems as they become available. Continue to expand the RS1 data files as new smoke components are identified. Enhance system management capability by a project members attendance at the Perkin-Elmer system managers course.

2. Continue to study cigarette models for visibility and related information as they become available.

3. Continue to investigate the distribution and quantitation of selected ingredients applied to cigarette models to enhance sidestream subjectives.

4. Supply GC and GC/MS analyses as required.

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PROJECT NUMBER: 2507
PROJECT TITLE: Radiochemical Investigations
PROJECT LEADER: S. B. Hassam
PERIOD COVERED: February, 1988

I. SIDESTREAM SMOKE

A. **Objective**: Develop methods for collection and radioanalysis of SS from cigarettes labelled with ^{14}C-glycerol.

B. **Results**: 1) Parts for a SAVA smoking machine are on back order. An alternative smoking and trapping assembly was set up, consisting of a single port smoking machine and impinger traps to collect SS and MS smoke components from the same cigarette. In one arrangement smoke is swept through two traps, and a Cambridge filter pad is placed at the end of the line to collect particulates. In a second arrangement the filter pad is placed first, followed by two traps to collect gas phase components.

Initially a smoking chamber of the type used for SAVA, was investigated. SS whole smoke from a 2R1 cigarette (nonradioactive) was swept first through two traps containing 2,4-DNPH solution to trap carbonyls, followed by a filter pad which was placed in 2,4-DNPH solution after smoking was completed. MS whole smoke was collected in the same way. RP-HPLC analyses of the smoke phases showed that this collection system was effective in trapping carbonyls of interest. The major carbonyl in the first SS trap was acetaldehyde, with lesser amounts of acrolein, acetone, propionaldehyde and formaldehyde. The second SS trap showed reduced amounts of acetaldehyde and acetone, with no detectable amounts of the other carbonyls. The SS pad extract contained some formaldehyde and acetone. The chamber was rinsed with 2,4-DNPH and RP-HPLC analysis showed formaldehyde to be the major carbonyl present. In the case of MS the first trap contained acetaldehyde, with lesser amounts of acrolein, acetone and propionaldehyde; the second trap and MS pad extract contained no detectable amounts of these carbonyls.

A chamber of the type used for the total recovery smoking machine was also investigated. This chamber is considered to be more amenable for use with radiolabeled cigarettes than the SAVA chamber. Three nonradioactive cigarettes were smoked: 2R1, 0% $Mg(OH)_2$ (code 3657) and 35% $Mg(OH)_2$ (code 3661). RP-HPLC analysis of smoke phases from the 2R1 and 35% ($Mg(OH)_2$ cigarettes showed essentially the same profile of carbonyls as described above. Data analysis of samples from 0% $Mg(OH)_2$ cigarettes is in progress.

Smoking experiments involving collection of particulate matter first, followed by solution trapping of gas phase components, have also been done. (RP-HPLC analyses are pending).

2. A third type of SS collection dome (originally used for CO/CO_2 analysis) was assembled, in conjunction with a single port syringe

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smoking machine. Sidestream components on the dome were trapped by rinsing with 2,4-DNPH after smoking was completed. Three nonradioactive cigarettes were smoked: 2R1, 0% $Mg(OH)_2$ and 35% $Mg(OH)_2$. In all three cases RP-HPLC showed formaldehyde to be the major carbonyl present.

A 2R1 cigarette spiked with ^{14}C-glycerol was smoked using the above setup. RP-HPLC and TLC radiochromatography is in progress.

3. Arrangements have been made with Radiomatic Instruments to complete assembly of the recently acquired gas radiochromatograph detector.

C. **Plans**: 1) Apply the impinger trapping system to collect smoke from ^{14}C-glycerol labeled cigarettes. Complete RP-HPLC of nonradioactive smoke phases. 2) Continue smoking experiments with ^{14}C-glycerol cigarettes. Complete radiochromatography analyses.

D. **References**: N.B. 8236, 8154, 8567, 8610.

II. SMOKE CHEMISTRY STUDIES (SIDESTREAM)

A. **Objective**: Determine total radioactivity distribution into smoke from ^{14}C-glycerol labelled cigarettes, using the total recovery smoking machine. Apply HPLC and TLC radiochromatography methods to analyze radiolabelled smoke phases.

B. **Results**: A memo was issued summarizing the total radioactivity distributions from ^{14}C-glycerol labeled cigarettes. In particular, it was observed for the three cigarette wrappers investigated - 2R1, 0% and 35% ($Mg(OH)_2$ - that a large percentage of the radioactivity is transferred to SS gas phase: 43.5, 48.9 and 58.4%, respectively. The ratio of activity in SS (gas phase + TPM + chamber wash) **vs**. MS (gas phase + TPM) was ca. 2 to 1 for 2R1 and 35% $Mg(OH)_2$ wrappers (higher porosity papers) and ca. 3 to 1 for 0% $Mg(OH)_2$ (lower porosity).

Radiochromatography analyses of smoke phases from 35% $Mg(OH)_2$ wrapper cigarettes (code 10-87-7) were completed, after derivatization with 2,4-DNPH. The radioactivity in SS TPM extracts was mostly ^{14}C-glycerol. The chamber wash (SS wash) showed ca. 2% of radioactivity to be ^{14}C-formaldehyde, the remainder being glycerol.

Radiochromatography of MS TPM and butt+ash samples was done for all 3 types of cigarettes. The radioactivity in these phases was >99% associated with ^{14}C-glycerol.

Of the carbonyls of interest the UV chromatograms showed formaldehyde, acetaldehyde and acetone to be present in SS TPM, chamber wash (SS), MS TPM, and butt+ash samples. No acrolein was detected in these phases.

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C. **Plans**: Prepare a summary of the radiochromatography analyses.

D. **References**: Memo from R. H. Newman to R. N. Ferguson, "Progress report on Sidestream Investigations", February 12, 1988.

III. OTHER ACTIVITIES

In collaboration with Project 2525, additional 2R1 filler was sprayed with ^{14}C-glycerol for preparation of radiolabeled cigarettes.

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PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
PROJECT LEADER: E. W. Southwick
PERIOD COVERED: February, 1988

I. GLUCOSE-DERIVED FLAVORANTS

A. **Objective**: To develop glucose-derived flavorants.

B. **Results**: 1. Menthol-glucose carbonate. Since establishing that two compounds in the mixture of compounds known as menthol-glucose carbonate are particularly efficient at delivering menthol to mainstream smoke, efforts have been directed at enhancing their proportion in the mixture. Modification of reaction parameters did alter the product distribution in the desired direction. More recently, an alternate acylating agent has been investigated.

Reaction of glucose and 1-menthyloxycarbonylimidazolide in either pyridine or dimethylformamide in the presence of about 20 mol% of 1,8-diazabicyclo[5,4,0]undec-7-ene produced a reaction mixture which consisted of mainly 6-O, 2-O- and β-1-O-menthyloxycarbonyl-D-glucoses, with the 6-O-isomer being the more abundant one. However there is an extra component which corresponds to a monocarbonate. The identity of this component is under investigation.

2. Glucose-vanillin. Reaction of vanillin with acetobromoglucose in aqueous acetone produced 4'-formyl-2'-methoxyphenyl 2,3,4,6-tetra-O-acetyl-β-D-glucopyranoside in 57% yield. Subsequent hydrolysis with methanolic NaOH gave glucovanillin in 90% yield. Currently about 10 gm of glucovanillin is on hand.

II. PROJECT EXTRA

A. **Objective**: To develop proprietary flavor additives for enhanced flavor perception in low delivery cigarettes.

B. **Results**: 1. CR-2643 (6-methylhexahydro-2(H)-benzofuranone) has been shown to evince desirable subjective properties. A structurally similar tetrahydrobenzofuranone has been synthesized by a two-step reaction sequence in overall yield of 27%.

2. The synthesis of dihydroactinidiolide, a tobacco-identical benzofuranone, following a literature report gave a disappointing yield. Subsequent modification of conditions has revealed the reaction to be sensitive to the acidic conditions. A 60% yield of a mixture of the desired compound and another compound, aeginetolide, has been obtained thus far. Optimization of reaction conditions is being pursued.

3. The acylation of 2,3-dimethylpyrazine with isovaleraldehyde gave a 54% yield of the expected ketone (black pepper, slight chocolate aroma). This ketone was cleanly reduced with sodium

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borohydride to give the corresponding alcohol (buttery, chocolate aroma). Several attempts to dehydrate this alcohol have been made; in most cases, the olefin was observed. However, the desired olefin is accompanied by varying amounts of disproportionation products.

4. Trimethylpyrazine was acylated with isovaleraldehyde to give the ketone in 67% yield. A portion of the ketone was converted to the hydrazone for subsequent reduction to the alkylpyrazine.

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PROJECT NUMBER: 2525
PROJECT TITLE: Tobacco Chemistry
PROJECT LEADER: S. Tafur
WRITTEN BY: R. Izac
PERIOD COVERED: February, 1988

I. NATURAL PRODUCTS CHEMISTRY

A. **Objective**: To isolate, identify and/or analyze natural compounds with major emphasis on tobacco and tobacco products.

B. **Results**: Additional quantities of Bright tobacco extract Fr IV were separated into four fractions by vacuum liquid chromatography (VLC) on ODS-silica. Analytical LC profiling of the VLC fractions on an aminopropylsilica column (Zorbax NH_2) was conducted.

C. **Plans**: Continue fractionation with emphasis on optimizing production.

D. **References**:
1. Tafur, S. Notebook No. 8417.
2. Core, M. Notebook No. 8608.

II. LOW NICOTINE

A. **Objective**: To assist in characterizing unextracted nicotine.

B. **Results**: It has been reported by several investigators that nicotine transfers into smoke without a change in optical purity. In order to verify this cigarettes were obtained and the nicotine isolated from the filler and from the smoke condensate. The general procedure for the isolation of nicotine involves extraction with 0.5N HCl, partition with dichloromethane, basification of the aqueous layer with KOH, partition with dichloromethane, evaporation of the solvent ($MeCl_2$, to yield the crude alkaloids), Kugelrohr distillation (to yield crude nicotine), and HPLC (silica, hexane/acetone/isopropanol/triethylamine, 65/30/5/1.5) to give purified nicotine. Prior to measuring the optical rotation, the nicotine was subjected to a final Kugelrohr distillation. The following yields of nicotine were obtained:

cig. #	quantity	wt. condensate (g)	wt. alkaloids (g)	wt. nicotine (crude, g)
5405	510	5.3	0.5	0.2
5406	3,000	29	2.5	0.2
5405*	500	---	0.8	0.5
5406*	1,500	---	0.4	0.1

5405 are control cigarettes with casing (filtered, 1.13mg nic/c).
5406 are ART cigarettes (filtered, 0.15mg nic/c).
*extraction of filler

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A reversed phase HPLC system for the determination of nicotine in aqueous solution is being optimized for a nicotine diffusion study. The lower limit of detection was found to be about 1 ng of nicotine per injection.

C. **Plans**: Continue measurement of optical rotations. Measure diffusion constant of nicotine. Start model study of the interaction of nicotine and protein.

D. **References**:
1. Izac, R. Notebook No. 8379.
2. Core, M. Notebook No. 8608.

III. GREENHOUSE STUDIES

A. **Objective**: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.

B. **Results**: Group nine, Burley 21 plants are being harvested at different intervals after topping to determine the optimum enzyme yields. The group ten plants have been set up and transplanted.

The total amount of dry weight ^{14}C plant material was 605g including 201g of leaf material. The average specific activity was 274µCi/g of tobacco. The amount of $^{14}CO_2$ injected into the chamber during Run #12 was 540.8 grams.

Total alkaloid and sugar analyses by AR have been completed for 158 S. C. Tests, 180 Speight Seed Farms 1987 Breeding lines, and 180 flue cured tobacco samples from the 1987 Northrup King Seed Co. Breeding line test.

Manicured 2R1 filler was sprayed with 500µCi of ^{14}C glycerol and a total of 34 hand made cigarettes were prepared.

A solution of sodium ^{13}C-β-methylvalerate was stem fed to a Coker 319 leaf. After uptake of the solution the leaf developed the aroma of β-methylvaleric acid. The leaf was oven dried and submitted for NMR analysis.

C. **Plans**: Continue to produce fresh root tissue by hydroponic culture. Isolate sucrose ester fraction from leaf fed β-methylvaleric acid.

D. **References**:
1. Bass, R. Notebook No. 8495.
2. Izac, R. Notebook No. 8379.

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IV. SS/MS FLAVOR DISTRIBUTION

A. **Objective**: Study the SS/MS distribution of vanillin/glucovanillin.

B. **Results**: Vanillin standard solutions were prepared and given to J. Naworal (Project 2501) for development of an analytical method. TPM pads were spiked with vanillin at three different levels and then extracted with acetone to determine extraction efficiency. Five PM Blues cigarettes were smoked on a SAVA smoking machine for the collection of MS and SS. Acetone extracts of MS and SS TPM pad, SS cold trap, SS chamber wash and butt with filter were obtained and provided to J. Naworal for analysis.

C. **Plans**: Continue smoking and analysis.

E. **References**: Tafur, S. Notebook No. 8490.

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PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: February 1988

I. PROJECT ART

A. **Objective**: To investigate the role and fate of ammonia, carbon dioxide, and pH in the ammonium bicarbonate (AB) application.

B. **Results**:

Time studies of the loss of carbon dioxide were conducted for thin bright and thin burley tobaccos. Generally, the measured amount of carbon dioxide on the tobacco after AB application never equalled the levels predicted by calculation. Within one hour after application the carbon dioxide levels were low but constant, even after two hours. A time study of ammonia in sealed and unsealed vials was conducted using thin bright tobacco and an AB application level of three percent. After eight days the ammonia values of the tobacco stored under these two conditions were essentially the same although somewhat lower than at day zero. This suggests that this tobacco, under these conditions, loses little ammonia, and what is lost is not lost due to volatilization.

A total of 50 tobacco samples were analyzed for nicotine content.

C. **Plans**: To continue the carbon dioxide and ammonia studies using other tobacco types.

II. ELEMENTAL ANALYSIS

A. **Objective**: To provide quantitative and qualitative elemental data on tobacco, cigarette paper, material evaluation samples, and special project samples.

B. **Results**:

The Tracor 4020 X-ray spectrometer was transferred to TQAF. It was recalibrated and restandardized at the new location. A bright monitor and it's specifications were sent as well. The X-ray spectrometer is ready for routine operation in QA.

The calibration and standardization of the Tracor 5000 X-ray spectrometer using the new thin-film software is complete. Known samples of cigarette and tipping paper are being analyzed to test accuracy. This software has potential applications in many areas of multielement analysis.

A total of 57 samples were analyzed for various elements in support of ART and other R&D projects. Samples of tipping paper were screened for relative elemental concentrations using X-ray fluorescence.

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C. **References**:

1. Lewis, J. Y., "Transfer and Maintenance of Tracor X-Ray Fluorescence Spectrometer," memo to D. Donher, February 1, 1988.

2. Lewis, J. Y., "X-Ray Fluorescence Analysis of Papers (request codes P883 and P885)," memo to R. Arthur, February 23, 1988.

III. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIALS

A. **Objective**: To provide headspace analysis for volatile solvents of various packaging materials.

B. **Results**: Several different capillary and megabore GC columns with thick films were investigated as alternatives to the Poraplot Q column currently in use. The major shortcoming of the Poraplot Q column is the shift in retention times of components as a function of amount present. None of the columns examined so far were judged as satisfactory, primarily due to incomplete retention or separation of some components in the headspace standard mixture.

C. **Plans**: Investigate other columns as possible replacements for the Poraplot Q. Continue to work on development of the overall method for transfer to QA.

IV. RESPONSE TO ANALYTICAL REQUESTS

A. **Objective**: To provide analytical support to R&D and Operations personnel and projects.

B. **Results**:

Analyses and investigations by the project personnel during the month of February included:

Four flavor samples were analyzed for various compounds at the request of FTR.

Two flavor samples were analyzed for CF. CF was present in one of the samples.

Two customer complaint samples were analyzed for possible contaminants. One sample contained high levels of tax stamp ink solvents.

C. **References**:

1. Ingraham, D. F., "CC 880203007, Control no. 8806 - Marlboro LS - Michael McGivney," memo to J. J. Pollard, February 13, 1988.

2. Ingraham, D. F., "PLC 88005, Control no. 8807 - Marlboro FTB - Michael Lorenzo," memo to J. J. Pollard, February 19, 1988.

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PROJECT NUMBER: 6906
PROJECT TITLE: Biological Effects of Smoke
PROJECT LEADER: J. M. Penn
WRITTENBY: B. D. Davies and W. R. McCoy
PERIOD COVERED: February, 1988

I. PROTEIN KINASE C WHOLE CELL ASSAY

A. **Objective**: To evaluate the dose response of phorbol-12,13-dibutyrate (PDBu) and 12-O-tetradecanoyl-phorbol-13-acetate (TPA) on the phosphorylation of specific membrane proteins of 3T3 cells examined at various stages of growth.

B. **Results**: Four experiments were performed in which ^{32}P labeled 3T3 cells were exposed to a range of PDBu (2.5-2520 ng/ml) or TPA (3-3080 ng/ml) concentrations for 1 min. or 15 min. Cells used were at various stages of growth: log phase, log phase with refeeding, "confluent" refed cells, and cells which were in log phase or "confluent" that were refed with low serum (0.5%) media to induce "quiescence." In addition to the standard assay protocol, some samples were run on a PhastSystem gradient and on a larger format gel system, both of which should improve the resolution of the subsequently produced autoradiographs. Quantitative analysis of autoradiographs produced from these gels cannot be completed until the laser densitometer software is received. However, qualitative analyses indicate that autoradiographs from this new Phastgel display enhanced resolution and should facilitate quantitative analytical measurements.

C. **Plans**: Experiments to systematically produce and evaluate specific cell stages and define conditions for their preparation are underway. These experiments include examining "log phase/quiescent" and "confluent/quiescent" cells using daily refeeding, and cell counts, protein measurements and microscopic examination as indicators of cell stage.

D. **Reference**:

G. M. Nixon. Notebook No. 8569, pp. 45-63.

II. BINDING OF 3H-PDBU TO 3T3 CELLS

A. **Objective**: Examine the effects of CSC on the binding of ^{3}H-PDBu to 3T3 cells.

B. **Results**: Subsequent analyses of the effects of CSC on ^{3}H-PDBu binding have indicated that CSC does not inhibit binding but rather stimulates it. Two additional experiments, which were performed with two different CSCs (a Kentucky reference control and 100% expanded stems), were completed this month and confirm this observation. When preincubated with cells for 19.5 hr, both CSCs stimulated ^{3}H-PDBu binding. TPA, used as a control in all CSC experiments, consistently inhibited binding. Due to the

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effects observed in the EGF assay, catechol was also tested. When catechol was added at time of binding, there seemed to be some inhibition of binding. However, preincubation of the cells with catechol for 19.5 hr. resulted in a stimulation of binding in one experiment and an inhibition of binding in a replicate experiment.

C. **Plans**: Repeat the preincubation experiments with catechol and include an examination of a dose response. Additionally, examine the effects of CSC on the kinetics of binding to determine if CSC effects the number of receptors per cell or just their affinity.

D. **Reference**:

T. J. Ferguson. Notebook No. 8572, p. 200.

III. PROTEIN KINASE C ASSAY

A. **Objective**: To determine the effects of CSC on isolated PKC activity.

B. **Results**: Several experiments were conducted this month and all failed to produce a dose response with CSC on PKC activity. Determinations of activity were complicated due to the existence of high background levels of stimulation caused by the solvent control.

C. **Plans**: Experiments have been designed which should lower the background levels of DMSO. These will be completed next month. In addition, modifications are being included which should lower CSC variability.

D. **Reference**:

B. G. Vaughan. Notebook No. 8537, pp. 87-105.

IV. INHIBITION OF EGF BINDING ASSAY

A. **Objective**: Establish the EGF assay and determine the effects of various CSCs on EGF binding.

B. **Results**: The contribution of catechol to the effects of 2R1 CSC was calculated using a published value for the concentration of catechol in 2R1 CSC (0.5% by weight). When the assumption is made that there is no interaction between catechol and other components in CSC, catechol could account for between 53 to 83% of the inhibition of EGF binding caused by 2R1 CSC and between 40 to 94% of the reduction in cell number. Two experiments were performed to determine the IC_{50} of catechol and its possible synergistic interaction with 2R1 CSC. Preliminary analysis of the data indicate that the IC_{50} for inhibition of binding by catechol and processed 2R1 CSC were, 0.77 ug/ml and 95 ug/ml, respectively.

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C. **Plans**: Obtain analytical data on catechol levels for CSC samples which have been tested in the EGF assay. Determine if catechol and 2R1 CSC interact synergistically. Examine the effects of fractions of 2R1 CSC.

D. **Reference**:

G. J. Patskan. Notebook No. 8609, pp. 1-176.

V. SALMONELLA/MICROSOME (S/M) ASSAY: EVALUATION OF MODEL CIGARETTES WITH STRAIN TA104

A. **Objective**: To evaluate the activity of five model cigarettes in the S/M assay using strain TA104.

B. **Results**: The rank order of activity (+S9) by increasing activity was 2R1 = RL Ref. < bright = burley = RCB. No new information was obtained from these results relative to other strains currently in use.

C. **Plans**: There are no plans to use TA104 for routine testing.

D. **Reference**:

L. Thompson. Notebook No. 8516, pp. 192-193.

VI. S/M ASSAY; SALT EFFECTS

A. **Objective**: To investigate the effect of the K/Ca ratio in filler on S/M activity by the addition of potassium acetate and/or calcium acetate to BuCel which is sprayed onto BrBW.

B. **Results**: No specific pattern of activity was seen from the K/Ca ratio experiments. As the % of calcium acetate added to the filler increased, the CSC yield decreased, and the S/M activity initially increased and then plateaued. However, at the highest calcium acetate level (low CSC yield), the S/M activity was significantly reduced. This finding may be related to smoking parameters and will be investigated further.

C. **Plans**: Additional experiments have been planned which include new controls which will assist in data evaluation.

D. **Reference**:

L. Thompson. Notebook No. 8516, pp. 192-193.

VII. S/M ASSAY; FILTER STUDIES

A. **Objective**: To investigate the effect of whistle through (WT control), charcoal, THAM on charcoal, or basic alumina filters on

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IT CSC from washed shredded bright stem (WSBS) and BCR blend cigarettes as measured by the S/M assay (TA98+S9).

B. **Results**: No difference in activity was seen as a result of the various filters. All of the WSBS IT CSCs were lower in S.A. than the BCR blend IT CSCs.

C. **Plans**: There are no immediate plans to evaluate additional filter types in the S/M assay.

D. **Reference**: L. Thompson. Notebook No. 8516, pp. 192-193.

VIII. GLUTATHIONE DEPLETION ASSAY (GDA): FILTER STUDIES

A. **Objective**: To investigate the effect of WT (control), charcoal, THAM on charcoal, or basic alumina filters on whole smoke (WS) from WSBS and BCR blend cigarettes as measured by the GDA.

B. **Results**: WS from charcoal, THAM on charcoal, and basic alumina filtered cigarettes (WSBS and BCR blend) was significantly lower in S.A. than WS from the WT control filtered cigarette (WSBS and BCR blend). The S.A. of the BCR blend cigarette WS was lower than the WSBS cigarette WS.

C. **Plans**: To repeat this study testing gas phase (GP).

D. **References**:

W. McCoy. Notebook No. 8484, pp. 108-195.
W. McCoy. Notebook No. 8585, pp. 36-61.

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PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate Studies
PROJECT LEADER: R. D. Kinser
PERIOD COVERED: February, 1988

I. TSNA PRECURSORS

A. **Objective**: To determine the precursors of MS TSNA.

B. **Results**: Mainstream delivery of nitric oxide on a per cigarette basis was higher for water washed oriental than for the starting material. The possibility that this relates to a nitrosating agent precursor which may be responsible for the increased NNK deliveries seen in other washed fillers will be investigated.

Eight model systems have been selected for consideration for further examination of the role of unextracted nicotine in TSNA pyrosynthesis.

A prototype of a device to measure puff-by-puff nitric oxide in smoke using the TEA as a detector was built and tested. Materials have been ordered to construct a model for routine use in TSNA precursor studies.

C. **Plans**: Evaluate results from nitrate addition and water washing experiments in light of recently acquired nicotine and minor alkaloid information. Determine appropriate model of unextracted nicotine and conduct add-back experiments to evaluate this model as a precursor of MS NNK. Determine the effect on TSNA production of adding NO to the smoke stream at the coal.

D. **References**:

Haut, S. A. Notebook No. 8595, p. 29.
Morgan, W. R. Notebook No. 8579, p. 32.

II. TSNA REMOVAL STUDIES

A. **Objective**: To explore the possibility of extracting TSNA from stored tobacco using an extraction fluid compatible with current processing.

B. **Results**: Experiments were conducted to evaluate the effects of an increased solvent:filler ratio and the effect of a one-hour equilibration time and lower flow rate on the extraction of TSNA and alkaloids from DBC burley with hexane. Analyses of alkaloid and TSNA content of extracted fillers are on-going.

It was determined that cotinine and NNN exhibit similar characteristics under some ion exchange conditions (cation exchange column (SPE); elution with 0.1M phosphate buffer, 10% ammonium hydroxide in 1:1 methanol:water, and 10% ammonium hydroxide in methanol). It appears that cotinine is a suitable model for TSNA

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in an evaluation of processes designed to remove alkaloids from an aqueous stream.

C. **Plans**: Continue investigation of cationic exchange SPE cartridges and/or cation exchange resins for TSNA analysis and removal of TSNA and alkaloids. Evaluate the analytical results and determine optimal hexane extraction conditions.

D. **References**:

Warfield, A. H. Notebook No. 8558, p. 61.

III. TSNA ANALYTICAL PROCEDURES

A. **Objective**: To develop and maintain analytical methodology for TSNA determinations which ensures optimal sample throughput, and precision and accuracy of results.

B. **Results**: Experiments were conducted to validate the use of the 20 g alumina column for preparation of samples enriched in VNA as well as TSNA. GC/TEA results indicated no VNA in the TSNA samples and no TSNA in the VNA samples. Quantitative agreement for first analyses was good for the TSNA, and acceptable for the purposes of these analyses for the VNA.

C. **Plans**: Perform replicate smokings and VNA analyses for a reference cigarette.

D. **References**:

Lambert, E. A. Notebook No. 8523, p. 110.

IV. CROSSED SOLUBLES/BASE WEB STUDY (CHEMISTRY)

A. **Objective**: To investigate the smoke chemistry of model cigarettes made from all possible combinations of solubles from bright, burley, and oriental tobaccos on base webs from the same three tobaccos.

B. **Results**: Cigarettes were prepared to investigate the effect of the potassium/calcium ratio on S/M activity and CSC delivery. Salts were dissolved in distilled water and added to Bu CEL, which was then sprayed onto Br base web. Increased calcium (added as calcium acetate) resulted in decreased TPM and significantly reduced sidestream visibility. Control K:Ca ratio was 1.58:1; K:Ca ratios of approximately 1:2 and 1:3 yielded the reduced TPM and sidestream visibility. Increased levels of calcium also resulted in collection of less impaction trapped CSC.

C. **Plans**: Additional experiments have been planned to replicate these results and to include more controls to assist in data interpretation.

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D. **References**:

Williams, D. L. Notebook No. 8530, p. 57.

V. OPTIMIZATION OF A LOW ACTIVITY MODEL: MS NITRIC OXIDE CONTENT

A. **Objective**: To develop methods of reducing MS NO delivery for a low activity model.

B. **Results**: Four types of filter treatments designed to reduce mainstream NO delivery have been identified: application of diene generators to the filters, addition of ferrous chelates to the filters, addition of spin traps to filters, and addition of oxidation/adsorption agents to filters. Materials have been ordered to begin evaluation of these methods. The necessary analytical equipment to determine NO deliveries with a nitrate selective electrode are also on order.

C. **Plans**: Validate analytical methodology and begin evaluation of the various filter additives.

D. **References**: Levins, R. J. Notebook No. 8550, p. 87.

VI. SUPPORT FUNCTION: CONDENSATE PREPARATION

A. **Objective**: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.

B. **Results**: Fifty-six smokings of 17 different cigarette codes were conducted to provide condensate for various chemical and biological assays.

Acid, base, and neutral fractions were prepared from condensate from two different codes for Dr. G. Patskan.

D. **References**:

Hellams, R. D. Notebook No. 8613, p. 8.
Lambert, E. A. Notebook No. 8523, p. 110.
Tickle, M. H. Notebook No. 8587, p. 81.

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PROJECT NUMBER: 0008
PROJECT TITLE: Computer Applications/Lab Systems
PROJECT LEADER: Mark Allred
PERIOD COVERED: February, 1988

I. GENERAL ANALYTICAL LIMS MIGRATION

A. **Objective**: Move General Analytical LIMS and data collection functions off of the HP 1000 LABSAM and LAS system.

B. **Results**: Gave GA access to GA LIMS Prototype. The "Hot Water Solubles" gravimetric workstation is near completion. When this unit is installed, the off-loading of gravimetric instruments from the HP 1000 will be completed. Ordered hardware and software to off-load chromatographic instruments.

C. **Conclusions**: Move ahead with off-loading the data collection and analysis functions from the HP 1000. Continue refining the GA LIMS Prototype.

D. **Plans**: Develop file transfer procedures between LABSAM on the HP 1000 and GA LIMS on the UNIX host. Finish the gravimetric area, including documentation. Test GA LIMS with complete dataset from LABSAM.

II. CTSD MODERNIZATION

A. **Objective**: Eliminate obsolete and difficult to maintain equipment in the CTSD area.

B. **Results**: Development required to eliminate dependency on the Honeywell Level 6 computer is completed. Equipment is on order to complete the installation. Dependency on the Decsystem 20 computer has been eliminated. A Functional Specification for the Gas Phase project has been generated by Development Engineering (DE) and is in review.

C. **Conclusions**: This is a long-term effort involving CAD, DE, and CTSD.

D. **Plans**: Finish Strip Chart Graphics application. Review Gas Phase Functional Specification. Develop design and implementation plans for this project.

III. FREEFORM SCREEN MANAGER AND BRITTON-LEE IDM DATABASE

A. **Objective**: Provide optimized database applications. Assist with checkout and troubleshooting of new releases.

B. **Results**: New Freeform version was not clean. It is much improved but still has problems. Set up Database Profile reports for PED

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and assisted with database load. Modified relief valve reports. CTSD Britton-Lee applications converted to use Ethernet TCP/IP.

C. **Conclusions**: The stability of these products needs to be improved.

D. **Plans**: Work on resolving known problems and on capacity and backup issues.

IV. LAB AUTOMATION

A. **Objective**: Assist scientists with specification of equipment needed for lab automation.

B. **Results**: Assisted three users with specification of computer equipment to do data collection and analysis from and control of lab instruments. Initiated two evaluations: TOPS/NFS and DADiSP.

C. **Plans**: Review specifications with user. Assist with procurement and installation. Evaluate TOPS/NFS and DADiSP.

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PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services Division
SECTION LEADER: Joyce F. Stargardt
PERIOD COVERED: February, 1988

I. MARKET ACTIVITY

A. **Objective**: To monitor and report the introduction of new brands and modifications to existing brands on the domestic and international markets.

B. **Results**:

1. Domestic

R. J. Reynolds has expanded the test market of Winston Lights with white tipping by introducing Winston Lights 85 with white tipping in Cleveland, Ohio. Winston Lights 100 cigarettes with white tipping were first test marketed in Cleveland, Ohio beginning December, 1987. The pamphlet accompanying these cigarettes says the reason for this change is that most people prefer white tipping on their low tar cigarettes. Winston Lights 85 and 100 are in national distribution with cork tipping. No changes were noted in the smoke deliveries between the white or cork tipped cigarettes.

2. International

Marlboro Lights King Size cigarettes have been introduced in the Japanese market. These cigarettes deliver 11 mg tar and 1 mg nicotine. These cigarettes are produced under a licensing agreement between Japan Tobacco, Inc. and Philip Morris.

R. J. Reynolds is exporting Island Lights King Size cigarettes to Japan. These cigarettes deliver 10 mg tar and 0.7 mg nicotine.

Japan Tobacco, Inc. is distributing Rivage 85 cigarettes with a triple aqua filter in the United Arab Emirates. This cigarette utilizes a "triple aqua-filter" which consists of an inner and outer cellulose acetate plug and a water-filled polyethylene capsule between the cellulose acetate plugs. The contents of the capsule are released into the smoke stream when the filter portion containing the capsule is squeezed on the blue line noted on the tipping paper. The advertising for this product claims: "you can enjoy smooth and refreshing flavor without harshness in the smoke thanks to the innovative absorption of smoke through the water-filled capsule." The TPM and water deliveries of this cigarette increase significantly after pinching the filter. The tar delivery is not affected by the release of the water because tar is defined as TPM-nicotine-water.

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	Rivage 85		
	Before Pinching	After Pinching	Advertised on Pack
Butt Length, mm	38	38	-
FTC Tar, mg/cigt.	11.5	12.3	11
TPM, mg/cigt.	13.9	18.3	-
Nicotine, mg/cigt.	0.66	0.72	0.6
Water, mg/cigt.	1.7	5.3	-
Puffs/cigt.	5.5	5.6	-

II. ANALYTICAL METHODS DEVELOPMENT AND SUPPORT

A. Objective: To evaluate and recommend analytical methods and new technology in support of programs for R&D and Manufacturing.

B. Results:

1. Coal Removal

The coal removal instrument has been modified to test ultra slim (17 mm circumference) cigarettes. These cigarettes have been analyzed for percent coal removal and the conclusion from the testing is that the ultra slim cigarettes have a higher coal retention than observed on the regular (25 mm) circumference cigarettes. This is probably related to the higher rod density of these ultra slim cigarettes.

2. Special Menthol Projects

The average menthol level of "inner" versus "outer" cigarettes was compared for three mentholated foil products - Marlboro Lights Menthol 85 (SP), Marlboro Lights Menthol 83 (Box), and Marlboro Menthol 85. Benson & Hedges Menthol 100 (SP) (conventional menthol spray application) was analyzed as a control. Samples were analyzed thirty and sixty days after manufacture. The results for the thirty day evaluation showed that the menthol level of the "inner" cigarettes was statistically lower (about 0.1 mg) than that of the "outer" cigarettes. This result was attributed to the slower migration of the menthol from the foil to the "inner" cigarettes because of their location in the pack. No statistical difference was observed for the control. For the sixty day evaluation, similar statistical differences were observed. However, for the Marlboro Lights Menthol 83 (Box), the menthol level of the "outer" cigarettes was statistically lower than that of the "inner" cigarettes. This result may be attributed to absorption of menthol from the "outer" cigarettes by the box. Although the menthol levels of the "inner" and "outer" cigarettes for the mentholated foil products were found to be statistically different in both the thirty and sixty day evaluations, a subjective difference may not be detectable.

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